STABILIZATION/SOLIDIFICATION OF SOIL CONTAMINATED WITH HAZARDOUS WASTES

BY

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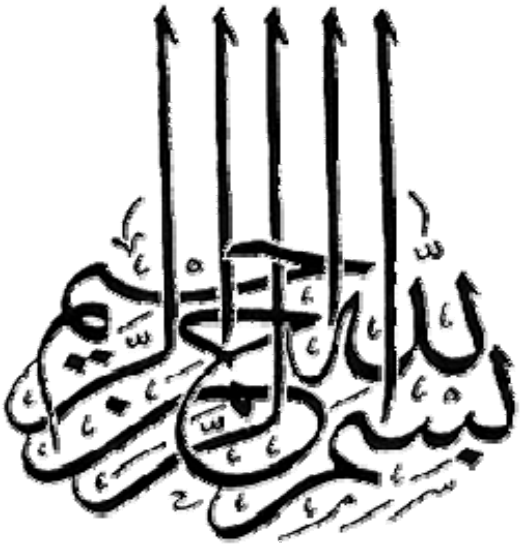
بسم الله الرحمن الرحيم

This work is dedicated to the memory of my father, my beloved mother, brothers and sister for all their love, encouragement and support. Also, to my wife, my jewel of inestimable affection

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

THESIS ABSTRACT

NAME: **LATEEF OLAWALE ALAMUTU**

TITLE: **STABILIZATION/SOLIDIFICATION OF SOIL CONTAMINATED WITH HAZARDOUS WASTES**

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Most available methods for the treatment of soils contaminated with industrial wastes are very expensive and result in products that require further treatment. Stabilization/solidification (immobilization) techniques, which are designed to decrease the leaching potential of heavy metals from soil by the addition of cement-based stabilizers/solidifiers, provide cost-effective solutions for soils contaminated with heavy metals.

In the present study, two types of locally available soils (marl and sand) were considered for synthetically contaminating with two types of potential wastes (electroplating and steel wastes generated from the local industries) and then stabilizing/solidifying (S/S) using plain cement (Type I ordinary Portland cement) and cement admixed with locally available fuel fly ash in varying dosages. The effectiveness of the S/S treatment was evaluated based on the density, unconfined compressive strength (UCS), porosity, permeability, leachability of the heavy metals, and the pH of leachates of the treated soil.

Results from the present experimental investigation indicate that the usage of ordinary Portland cement (OPC) with fuel fly ash (FFA) as stabilizers/solidifiers for soils contaminated with the heavy metals is more effective in reducing the laecahbility as compared to S/S using OPC alone. However, partial replacement of OPC by FFA in the binder system reduces the density, UCS, porosity as well as the permeability of solidified samples. Results also indicate that the leachability of the heavy metal in the treated sand soil is less than that in the treated marl soil.

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ملخص الرسالة

الإســـــــــــم : لطيف أولاويل ألماتو

عنوان الرسالة : تثبيت وتصليب التربة الملوثة بمخلفات خطرة

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تعتبـر معظـم الطـرق المتاحـة لمعالجـة التربـة الملوثـه بمخلفـات صـناعية عاليـه التكلفـة بالإضـافة الـى تسـببها فـي منتجـات تحتـاج الـى معالجـات اضـافية. تصـمم تكنلوجيـا التثبيـت والتصـلب لتقليـل الترشـح المحتمـل للمعـادن الثقيلـة مـن التربـة وذلـك عـن طريـق إضـافة الاسـمنت كمـادة مثبتـة لتقـديم حلـول مناسـبة الكلفـة للتـرب الملوثـة بالمعادن الثقيلة.

فـى هـذة الدراسـة، تـم اعتبـار نـوعين مـن التربـة المتـوفرة محليـا (الرمـل والتربـة الجيريـة والتـي تسـمي محليـاً بالمـارل) ملوثـه صـناعيا بنـوعين مـن المخلفـات المحتملـه وهمـا مخلفـات الطـلاء ومخلفـات الحديـد والمتولـدة مـن الصـناعات المحليـة. وقـد تـم تثبيتهمـا بإسـتخدام الأسـمنت البورتلانـدى النـوع الأول والأسـمنت المخلـوط بالرمـاد النفطـى المتطـاير بنسـب مختلفـة. كمـا تـم تقيـيم فعاليـة الثبيـت والتصـلب اعتمـادا علـى الكثافـة، والانضـغاطية الغيـر محصـورة، والمسـامية، والنفاذيـة، وقابليـة الترشـيح للمعـادن الثقيلـة، ونسـبة الهيـدروجين (pH) للمرشح لكلا التربتين المعالجتين.

أشـارت نتـائج التجـارب المعمليـة الـى أن أسـتخدام الأسـمنت البورتلانـدى مـع الرمـاد النفطـى المتطـاير كمثبـت ومصـلب للتـرب الملوثـة بالمعـادن الثقيلـة أكثـر فعاليـة فـى التقليـل مـن قابليـة الرشـح مقارنـة للتثبيـت والتصـلب باسـتخدام الأسـمنت البورتلانـدى بمفـرده. مـع أن الأسـتبدال الجزئـى للأسـمنت البورتلانـدى بالرمـاد النفطـى المتطـاير يقلـل مـن الكثافـة، وقـوة الإنضـغاطية الغيـر محصـورة، والمسـامية بالإضـافة الـى النفاذيـة للعينـات المتصـلبة. كمـا أثبتـت النتـائج أن رشـح المعـادن الثقيلـة فـى التربـة الرمليـة المعالجـة أقـل منهـا فـى تربـة المـارل المعالجة ايضا.

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CHAPTER ONE

INTRODUCTION

1.1 STABILIZATION/SOLIDIFICATION OF SOIL CONTAMINATED WASTES

Rapid industrialization of the developing countries calls for proper management of industrial wastes. Most industrial wastes are hazardous thereby having a far reaching impact on the environment if these wastes are not properly disposed off. These wastes directly or indirectly contribute massive amounts of hazardous contaminants into the environment of which the soil surface and subsurface are among the recipients. These soil contaminants pose serious environmental and health hazards, particularly to young children of which lead poisoning are prevalent among other heavy metal industrial wastes (Moon and Dermatas (2006). These waste materials are often dumped together with non-hazardous waste in the industrial landfill, which are not normally designed to handle hazardous waste materials. Hazardous waste materials commonly found in aqueous solution or suspension often require pretreatment before land-filling.

The presence of contaminated and abandoned land or 'brownfields' is an issue of great concern in many industrialized nations of the world, particularly in the United States and European Union (Yin et al., 2007). Soil contamination by hazardous wastes generated from various industries is reported to be one of the serious problems of environmental pollution. Industrial activities have left hectares of land contaminated with

detrimental chemicals that render the land unusable for future development. Due to shortage of uncontaminated sites or 'greenfields', developed nations have adopted several methods of reclamation of contaminated sites to facilitate sustainable industrial development, such as soil washing, stabilization/solidification, etc. For preventing soil contamination by hazardous wastes, several methods of disposal and treatment have also been adopted.

Among several soil waste remediation techniques that are being used, stabilization/solidification (S/S) method seems to be more effective because it binds the compounds of hazardous waste stream into a stable insoluble form (i.e., stabilization) or entrapping the waste within a solid cementitious matrix (i.e., solidification) (Wiles, 1987). The S/S technology had been described by the U.S. Environmental Protection Agency (EPA) as the best available technology for various hazardous wastes listed under 57 RCRA (Resource Conservation and Recovery Act) (Paria and Yuet 2006).

Stabilization refers to those techniques which reduce the hazard potential of a waste by converting the contaminants into their least soluble, mobile or toxic form. The physical nature and handling characteristics of the waste are not necessarily changed by stabilization (US Army Corps of Engineer, 1995).

Solidification refers to techniques that encapsulate the waste in a monolithic solid of high structural integrity. Solidification changes the physical properties of a contaminated substance. The desired changes include: increase in compressive strength, decrease in

permeability, and encapsulation of hazardous constituents (US Army Corps of Engineer, 1995). The encapsulation may be of fine waste particles (micro-encapsulation) or of a large block or container of waste (macro-encapsulation). Solidification does not necessarily involve a chemical interaction between the waste and the solidifying reagents, but may mechanically bind the waste monolithically. Contaminant migration (i.e., leaching) is restricted by decreasing the surface area exposed to leaching and/or by isolating the waste within an impervious capsule. Rain or other water cannot pick up or dissolve the chemicals as it moves through treated soil.

There are two basic types of S/S reagents, organic and inorganic (US Army Corps of Engineer, 1995). However, inorganic reagents are mostly used. The normal processing steps when using inorganic reagents are to

(1) Chemically react with all the water present,

(2) Chemically react with the contaminants to render them insoluble, and then

(3) Encapsulate the products.

Inorganic reagents most often used for S/S include Portland cement, fly ash, lime, phosphates, and kiln dust from lime and cement production, blends of cement and fly ash, and blends of lime and fly ash. All of these reagents are refereed as additives or binders in literature. They have basically the same general types of active ingredients as far as S/S reactions are concerned. These active ingredients include CaO, SiO_2, Al_2O_3, Fe_2O_3, and MgO. Organophillic clay had also being suggested for use due to the fact that they can absorb organic contaminant and trap it into a solid matrix. However, strength is

adversely affected when using organophillic clay (US Army Corps of Engineer, 1995). Therefore, its usage might not be considered for the project at hand since the reuse of the S/S soil is our major concern. It is also reported that the coarse-grained waste stabilized with quicklime or other reagents have poor geotechnical or environmental properties due to the coarse-grained limited pozzolanic surface area available for cementing, so their reuse in construction application might not be possible but with the addition of fly ash, their pozzolanic surface area can be increased (US Army Corps of Engineer, 1995).

S/S refers to treatment processes that are designed to accomplish one or more of the following:

- Improved handling and physical characteristics of the waste,
- Decreased surface area of the waste mass across which loss of contaminants can occur, and
- Reduced solubility of hazardous constituents in the waste.

S/S is a cleanup technology which involves mixing of contaminated soil generally with cement-based binders like Portland cement, lime/fly ash, and cement/fly ash, as mentioned earlier, so as to immobilize the contaminants within the soil by chemical and physical reactions which reduce leaching of the contaminants to the environment causing groundwater pollution (Paria and Yuet 2006). It is a process that physically encapsulates the contaminants.

S/S does not remove the contaminants from the soil but it only immobilizes the contaminant from having access to the environment. The S/S process has found applications in the treatment of liquids, soils and sludge contaminated with heavy metals and inorganic materials but may not be employed for organically contaminated soil due to their volatility and interference with the reagent setting process. Methods for studying the effectiveness of S/S process are physical, chemical and micro-structural. The degree of effectiveness of the soil treated by S/S technique is assessed mainly by the strength, permeability and leaching resistance of the treated soil.

S/S treatment can either be in situ or ex situ. In-situ process involves injecting reagents into the ground and mixing the reagents and the contaminated soil with an auger while the ex situ process consists of a pug mill mixer, chemical storage and feed devices, pumps, conveyors, and metering and measuring equipment.

Post treatment testing of the S/S process is both chemical and physical and may vary from project to project. The required chemical test is Toxicity Characteristics Leaching Processes (TCLP) and chemically analyzing the extract while the physical testing involves the unconfined compressive test, durability test and the permeability test. In addition to these tests, XRD and scanning electron microscope (SEM) determination to clarify the underlying mechanisms of contaminant immobilization.

Some of the advantages of the S/S technology over other remediation technologies are as follows (Paria and Yuet 2006):

- Good long-term stability, both physically and chemically,

- Good impact and comprehensive strength,
- High resistance against biodegradation,
- Relatively low water permeability, and
- Non-toxicity of the chemical ingredients used for S/S

In the Eastern Province of the Kingdom of Saudi Arabia, there are many industries located in Dammam industrial areas and the industrial city of Jubail. Among various types of industries, there are industries such as electroplating and steel industries which constitute about 10% of the total number of industries. Although data on the amount and composition of hazardous wastes locally generated are not available, it is a matter of fact that such industries generate a huge amount of hazardous wastes posing serious problems of soil contamination which eventually results into ground water pollution. There is no information available regarding soil pollution due to dumping of untreated industrial hazardous wastes on ground but the possibility of such things can not be ruled out. Hence, there is a need to come forward with a remedial measure well in advance in case the need for the treatment of contaminated soil arises. Keeping this in mind, it is highly desirable to explore various possibilities of effective and economical S/S treatment of local soils contaminated with some of the potentially hazardous wastes such as wastes from electroplating and steel industries.

1.2 NEED FOR THE RESEARCH

A survey of Dammam industrial area shows that steel and electroplating industries constitute about 10% of the total number of the industries in the area. Attempts were made to collect information pertaining to composition and quantity of the wastes

generated from these two types of industries which fall under potential sources of hazardous wastes containing heavy metals. Although no information were available from the local sources, following information were collected from literature.

- Typical examples of heavy metals found in wastes from steel and electroplating industries:
    - Steel industry wastes contain Fe, Mn, Hg, Ni, Pb, Cd, Cr, Cu, Co, and Zn (Brigden, et al., 2000).
    - Electroplating wastes contain Cr, Pb, Cd, Cu, Zn, and Fe (Asavapisit and Chotklang, 2004).
- Typical examples of the quantities of wastes generated from steel and electroplating industries:
    - 268,300 tons of steel foundry dust was produced in Turkey in a year (Salihoglu et al 2007).
    - 32,500 tons of soil contaminated by Genzale Plating Company, New York (EPA NPL listing, 1987).

The S/S technology for treating the contaminated soil using cementitious materials is the most suitable approach. However, applications of S/S technology for treatment of soils contaminated with hazardous wastes are not reported in context to Saudi Arabia, although like other countries, there is also a high possibility of soil and groundwater pollution due to dumping of various types of hazardous wastes on ground without treatment.

The need for the present study was realized considering the following major points:

- Although the S/S technology is a well known and established means of treating and reclaiming the contaminated soils, no research works on of S/S technology have so far been reported from KFUPM in particular and from Saudi Arabia in general and, therefore, there is need to assess the effectiveness of S/S treatment on local soils.

- Several pozzolanic waste products had been effectively used as a part replacement of OPC in S/S treatment. Since fuel fly ash (FFA) is generated in abundant quantities in Saudi Arabia, it is a matter of great interest to explore possibility of using FFA as partial replacement of OPC in S/S treatment of local soils contaminated by the selected wastes.

1.3 RESEARCH OBJECTIVES

The primary objective of this study is to evaluate the effectiveness of OPC-FFA based S/S treatment of two types of local soils (sand and marl) artificially contaminated with two types of synthetic industrial hazardous wastes (steel and electroplating industries wastes) in terms of density, unconfined compressive strength (UCS), porosity and permeability as well as chemical properties such as leachability of the metals and TCLP analysis of the treated soils.

The specific objectives are as follows:

- To optimize the dosage of fuel fly ash (FFA) to be used as part replacement for Portland cement in S/S treatment
- To optimize the moisture content for S/S treatment
- To optimize the dosage of ordinary Portland cement (OPC) for S/S treatment of the selected local soils, contaminated with electroplating and steel industries' wastes

1.4 THESIS ORGANIZATION

The detailed literature review is presented in the chapter two, which includes soil contamination by hazardous wastes, stabilization/solidification technology, tests carried out to measure the effectiveness of S/S technology and treatment of contaminated soils using S/S technology. The chapter three is devoted to the preliminary experimental investigation carried out to select the optimal levels of variables that will be used in the detailed experimental program. In chapter four a methodology for detail experimental program is presented. Results and discussions are presented in chapter five. Summary, conclusions and recommendations for further studies are presented in chapter six.

CHAPTER TWO

LITERATURE REVIEW

2.1 SOIL CONTAMINATION WITH HAZARDOUS WASTES

LaGrega et al (2001) defined hazardous wastes as waste (solids, sludges, liquids, and confined gases) other than radioactive (and infectious) wastes which, owing to their chemical activity or toxic, explosive, corrosive, or other characteristics, cause danger or likely to cause danger to health or the environment, whether alone or when coming into contact with other wastes.

Soil contamination is caused by the presence of man-made chemicals or other alteration in the natural soil environment. It is either liquids or solids hazardous substances mixed with soil. Usually, contaminants in the soil are physically or chemically attached to soil particles, or, if they are not attached, are trapped in the small spaces between soil particles. Soil contamination results when hazardous substances are either dumped or buried directly in the soil or migrate to the soil from a spill. Contaminants in the soil adversely affect plant growth and the human health. Moon and Dermatas (2006) reported that soils contaminated by lead have a far reaching health effect on young children. Protecting the environment from hazardous waste pollutants associated with waste generation and improper disposal is a major concern in today's industrialized world especially in the developing nations, thus there is a need to stabilize the hazardous waste in the contaminated soil to a nontoxic form or reduce the potential release of toxic species

into the environment. A well known case of soil contamination that affected human population was the Love Canal in Niagara Falls, in the United States.

Several waste management methods that are currently being used in treating hazardous wastes include: *selective ion exchange* for treating metal plating wastes (Leinonen, 1999); *thermal remediation* by introduction of heat to raise subsurface temperatures sufficiently high to volatize chemical contaminants out of the soil for vapor extraction **(**JoAnn et al. 1990**;** Roach et al. 2009; Pedron et al. 2009; Navarro et al. 2009**)**; *in-situ biological treatment* method which involves biodegradation of organic wastes **(**Lebeau et al. 2008; Fornes et al. 2009; Kumpiene et al. 2009**)**; *stabilization/solidification* for treating heavy metal wastes and other organic wastes (Katsioti et al. 2008; Moon et al. 2009; Chen et al. 2009; Choi et al. 2009; Zhang et al. 2009; Dermatas and Meng 2003; Yin et al. 2006; Yin et al. 2007; Moon et al. 2008; Moon et al. 2009). Out of all these treatment methods, the stabilization/solidification (S/S) technology has proven to be a best demonstrated available technology (BDAT) and it's less expensive.

2.2 STABILIZATION/SOLIDIFICATION (S/S) TECHNOLOGY

This technology was originally developed in the 1950's, but only recently has been used as treatment for industrial, hazardous and some selected radioactive wastes. Wiles (1989) defined S/S as treatment processes designed to improve waste-handling and physical characteristics, decrease surface area across which pollutants can transfer or leach, or limit the solubility or reduce the toxicity of hazardous constituents. The S/S technology had been adopted by governmental agencies and individuals and it is found to

be reliable. The S/S process consists of addition of cementitious binders like lime or cement or blends of cement and mineral admixtures (fly ash, silica fume, granulated ground blast furnace slag, etc.) to waste materials in form of slurry or liquid waste mixed with soil in predetermined proportions so that the contaminated soil will be stabilized/solidified preventing the waste from polluting the groundwater and subsequently the environment (Conner, 1990; Dermatas et al, 1996; Parsa et al, 1996; Janusa et al, 1998; Sanchez et al, 2000; Stegemann and Buenfeld, 2003; Randall and Chattopadhyay, 2004; Malviya and Chaudhary, 2006, Dermatas and Moon, 2006; Schifano et al, 2006; Yin et al, 2007). *Stabilization* is a process by which waste is converted to a more chemically stable form thereby reducing the hazard potential of waste. There is chemical reaction between the wastes and the binders added to reduce the contaminant of concern to their least soluble, mobile and/or toxic state (Wiles, 1989) while *solidification* is the technique that encapsulate the waste in a monolithic solid of high structural integrity, it may or may not involve a chemical reaction or chemical bonding with the waste contaminants and the solidifying reagents (Rho et al, 2001). Contaminant migration is restricted by decreasing the surface area exposed to leaching and/or by isolating the wastes within an impervious capsule.

2.2.1 Mechanism of S/S Technology

Immobilization of contaminants using the S/S technology involves either physical or chemical mechanisms. Physical stabilization changes the physical form of the waste but does not necessarily cause chemical binding of the waste constituents, while the chemical stabilization changes the chemical states of waste constituents to forms with lower

aqueous solubilities (Means et al., 1995). Most contaminants in S/S technology are immobilized by the physical mechanism and it involves macro-encapsulation; micro-encapsulation and embedment.

Macro-encapsulation involves the physical entrapment of contaminants in a large structural matrix (Pollard and Hills, 1987). Contaminants are held in a discontinuous pore within the stabilizing materials. Mohamed et al (2007) stated that the degree of macro-encapsulation is usually enhanced by the type and energy per unit mass of mixing. This they observed will prevent the breakdown of stabilized mass due to imposed environmental stresses (repeated cycles of freezing and thawing; introduction of percolating fluids; and physical loading stresses).

Micro-encapsulation is a process where the contaminants are entrapped in the pore spaces of solidified matrix. The contaminants and the matrix become homogenous after a long term. Micro-encapsulation is more susceptible to contaminants released from the stabilized mass if there is breakdown of the material due to decrease in particle size thereby exposing more surface area. High energy per unit mass in mixing must be maintained.

Embedment involves incorporation of large masses of contaminants into a solid matrix before disposal. Yilmaz et al (2003) stated that embedment is used in situations where it is impractical to reduce the bulk of the waste but where the waste is hazardous enough to be treated prior to disposal.

2.2.2 Wastes Treated using S/S Technology

The types of waste for which the S/S technology is used include either waste sludge from industries or artificially engineered waste in the laboratory. Heavy metals and their compounds are the major contaminants treated by the S/S technology due to the fact that metal contaminants cannot be destroyed by chemical, biological or thermal methods. Yin et al (2007) stated that there is need for the heavy metals to be extracted from the contaminated soil through soil washing or through immobilization using S/S process. Treatments of organics contaminants such as heavy sludges or soil contaminated with organics are amenable to S/S technology. Yilmaz et al (2003) observed that using cement as high as 35% on polychlorinated biphenyl (PCB) organic wastes, the leachates concentrations were above the regulatory standards same trend was observed when cement was used in stabilizing pulp and paper sludge containing adsorbable organic halogen (AOX), the conclusion was to use cement at higher percentage and other additives such as industrial adsorbents with Portland cement. The technology had also been adopted for the cleaning up sites contaminated by radioactive materials (i.e., reclamation of waste land).

Malviya and Chaudhary (2006) observed that cement-based S/S is best for inorganic wastes; especially those containing heavy metals. Metals are retained in the form of insoluble hydroxide or carbonate salts within the hardened structure as a result of the high pH of cement suited for insolubility of the heavy metal wastes. But for organic contaminants, there is interference with hydration process, reducing the final strength and

they are not easily stabilized. Cement also help in neutralizing the acidic waste due to its high alkalinity.

2.2.3 Binders used in S/S Technology

Binders used in the S/S technology are either inorganic (lime or cement and pozzolanic materials) and the organic binders (thermoplastic and thermosetting organic polymers). Table 2.1 (LaGrega et al; 2001) shows reagent applicability for waste stabilization. The optimum mix ratio for reagent to waste (i.e., contaminated soil) is typically around 0.25 (by mass), however, it can still vary in the range of 0.1 to 2.0 (US Army Corps of Engineer, 1995).

2.2.3.1 Inorganic binders

The two principal types of inorganic binders are cement binders and the pozzolanic binders (lime, kiln dust, fly ash). Janusa et al (1998) stated that a pozzolan is a material containing silica or silica and alumina that has little or no cementation value itself but, under some conditions, can react with lime in the presence of water to produce cementitious material. These pozzolanic binders are rarely used alone but commonly used as mineral admixtures in cement.

Cement as binder

Use of cement as binder is an established technology for the stabilization of hazardous waste and in the remediation of contaminated sites. Several researches in stabilizing and solidifying hazardous waste had employed cement as the main binder due to its relative availability, low cost, well-known setting and hardening reactions and some

existing data on the immobilization of metals (Means et al., 1995; LaGrega et al., 2001; Vipulanandan and Wang 1999). Cement help in reducing the mobility of contaminants, minimize free liquids and help in increasing strength of the contaminated soil. The main constituent of cement that gives strength is the tri- and dicalcium silicates ($3CaOSiO_2$ and $2CaOSiO_2$, abbreviated as C3S and C2S) which in the presence of water hydrate to form calcium- silicate-hydrate (C-S-H) gel (LaGrega et al; 2001) giving strength or tobermorite gel and crystalline calcium hydroxide (Yin et al 2007). Strength and permeability are affected by the water/cement ratio used in mixing because water/cement ratio affects the porosity of the solidified mass which controls both the strength and permeability. Wang and Vipulanandan (2000) observed that about 0.48 of water to cement (w/c) ratio, the cement will fully hydrate. Lee and Haas (1995) used cement on a brownfield site and the site was subsequently developed into a field.

Table 2.1 Reagent applicability for waste stabilization (LaGrega et al; 2001)

Waste component	Cement-based binders	Pozzolan-based binders	Thermoplastic binders	Organic polymer binders
Nonpolar organics as oil and grease, aromatic hydrocarbons, halogenated hydrocarbons, PCBs	May impede setting. Decreases durability over a long time period. Volatile may escape on mixing. Demonstrated effectiveness under certain conditions	May impede setting. Decreases durability over a long time period. Volatile may escape on mixing. Demonstrated effectiveness under certain conditions	Organics may vaporize on heating. Demonstrated effectiveness under certain conditions	May impede setting. Demonstrated effectiveness under certain conditions
Polar organics as: alcohols, phenols, organic acids, glycols	Phenol will significantly retard setting and will decrease durability in the short run. Decreases durability over a long time period	Phenol will significantly retard setting and will decrease durability in the short run. Alcohols may retard setting. Decreases durability over a long time period	Organics may vaporize on heating	No significant effect on setting
Acids as: hydrochloric acid, hydrofluoric acid	No significant effect on setting. Cement will neutralize acids. Type II and IV Portland cement demonstrate better durability characteristics than Type I. Demonstrated effectiveness	No significant effect on setting. Compatible, will neutralize acids. Demonstrated effectiveness.	Can be neutralized before incorporation	Can be neutralized before incorporation. Ureaforaldehyde demonstrated to be effective
Oxidizers as: sodium hypochlorate, potassium permanganate, nitric acid, potassium dichromate	Compatible	Compatible	May cause matrix breakdown, fire.	May cause matrix breakdown, fire.
Salts as: sulfates, halides, nitrates, cyanides	Increase setting times. Decreases durability. Sulfates may retard setting and cause spalling unless special cement is used. Sulfates accelerate other reactions.	Halides are easily leached and retard setting. Halides may retard setting, most are easily leached. Sulfates can retard or accelerate reactions.	Sulfates and halides may dehydrate and rehydrate, causing splitting	Compatible
Heavy metals as: lead, chromium, cadmium, arsenic, mercury	Compatible. Can increase set time. Demonstrated effectiveness under certain conditions.	Compatible. Demonstrated effectiveness on certain species (lead, cadmium, chromium).	Compatible. Demonstrated effectiveness on certain species (copper, arsenic, chromium).	Compatible. Demonstrated effectiveness with arsenic
Radioactive materials	Compatible.	Compatible.	Compatible.	Compatible.

Pozzolanic materials as binders

Yousuf et al (1995) defined pozzolan as a material that react with lime in the presence of water to produce a cementitious material. Common examples of pozzolans are fly ash, ground granulated blast furnace slag, cement kiln dust and pumice. The pozzolan contains a high amount of silicates and oxides of metals, due to less amount of calcium oxide which gives strength properties in the case of cement, the pozzolan are rarely used as main binder, but in combination with cement or lime they perform well. Dermatas el al (2003) used a combination of fly ash and lime in the S/S of soil contaminated with heavy metal and observed that the formation of calcium aluminate sulphate hydrate or ettringite was responsible for strength.

The use of fly ash as a part replacement for cement reduces the cost of S/S technology since the fly ash is a waste product generated by burning coal or other fuel. Several types of fly ashes have been used for S/S. Yin et al (2006) used up to 30% of rice husk ash as replacement of OPC. The strength obtained after 28 days of curing satisfied the landfill condition. Yin et al (2008) used oil palm ash as replacement in the S/S treatment of nickel hydroxide. They found that 20-30% replacement did not hardened after 28 days due to less CaO but concluded that using 15% replacement gave satisfying results. Asavapisit et al (2005) used pulverized fuel ash containing 12.94% of CaO as replacement in the treatment of plating sludge, the highest strength was observed from cement blended with 20 % PFA (by mass) during the 29 and 91 days of curing. This was due to the calcium hydroxide crystal produced during OPC hydration was consumed during the pozzolanic reaction of PFA, which occurred mostly during these curing

durations, and as a result, the secondary calcium silicate hydrate (C-S-H) gel was formed. It is believed that this secondary C-S-H gel contributes to the additional strength and reduced permeability for the blended cement paste other than the primary C-S-H gel obtained from the hydration of OPC alone.

The main product of hydration giving the early strength and stability is ettringite $[Ca_3Al(OH)_6]_2(SO4)_3.26H_2O$, this has been found to be responsible for high strength and low swell of the treated solids. Some factors affect strength development in solidification recipes and they include water. With increase in the quantity of water, there will be a decrease in the strength, also the smaller the waste to binder ratio, the better the physical and chemical properties of the solidified monoliths. Increased in strength can also be attributed to increase in the amounts of clay alumina and silica The strength tests for experiments carried out by Qian et al (2006) on S/S treated industrial waste sludge using Municipal Solid Waste Incineration (MSWI) fly ash admixed with cement at varying percentages of the cement and MSWI fly ash. It was found out that using 50% MSWI fly ash with no cement gives strength of 0.36 MPa (52 psi) while with 5% cement alone a similar strength was measured after 3 days. This is as shown in Table 2.2 for different combinations of cement and MSWI fly ash, comparison was made between 3 days and 7 days.

Table 2.2 Compressive strength of different mix proportion (Qian et al., 2006)

Sludge mix	3-Day compressive strength (MPa)	7-Day compressive strength (MPa)
A2	0.12	0.35
A3	0.16	0.22
A4	0.61	0.99
A5	0.17	0.15
A6	1.31	2.05
A7	0.90	1.31
A8	1.94	2.05
B3	0.03	0.03
B4	0.06	0.07
B5	0.05	0.04
B6	0.03	0.03
B7	0.05	0.04
B8	0.03	0.03
C3	a	a
C4	0.29	0.67
C5	a	a
C6	0.18	0.52
C7	0.1	0.14
C8	1.11	1.76

[a] Sample disintegrate upon demoulding from cast (due to very low strength)

From the above table, sludge mix A contain Cu and Pb-based sludge, while the sludge mix B contain Zn and Mn-based sludge and the sludge mix C are Ni-based sludge. The sludges were mixed with different proportions of cement and MSWI fly ash according to Table 2.3

Table 2.3 : Different mix proportions (wt. %) (Qian et al., 2006)

Mix	OPC (%)	MSWIFA (%)	Sludge (%)
1	0	0	100
2	0	50	50
3	5	0	95
4	5	45	50
5	10	0	90
6	10	45	45
7	15	0	85
8	15	40	45

OPC Ordinary Portland cement; MSWIFA Municipal Solid Waste Incineration Fly Ash

Solidified products are expected to harden over 2-5 days period. But there was strength loss up to 99% in an experiment with 3% (w/w total solids) of zinc containing metal plating waste after 28 days of curing compared to 1% (w/w total solids) of same waste using same OPC binder tested after 3 days (Pollard and Hills, 1997). Same trend was reported by Qian et al. (2006) comparing the strength after 3 day of curing and 7 days of curing with increasing waste content. Other factors that affect the strength development of the S/S product waste/binder ratio and water/binder ratio. Malviya and Chaudhary (2006) reported a waste/binder ratio of 0.4-0.5 and water/binder ratio of 0.4-0.6 will be adequate for strength.

2.2.3.2 Organic binders

Use of organic binders is still limited and very few have been successful due to reduction in strength of the solid mass. The organic binders that have been tested include asphalt (bitumen), polyethylene, polyesters, urea formaldehyde and polyolefin encapsulation (LaGrega et al; 2001 and Means et al; 1995). Organophillic clay (US Army Corps of Engineer, 1995) and activated carbon (Yukselen 2001) have been used. Organic binders have also found usage in treating radioactive wastes and hazardous organics that cannot be destroyed thermally. Organic binders are reported to deteriorate due to environmental factors like biological action or exposure to ultraviolet light (Means et al; 1995). S/S processes using organic binders are of two types. Thermosetting processes which involve formation of polymer to immobilize wastes, resulting into low density material, and requiring low density of additives. However, it involve a high cost so its not suited for site remediation processes (Lo, 1996). The other type of the organic binder used in S/S processes is the thermoplastic binders which involve stabilizing waste by

blending molten thermoplastic materials with wastes at high temperatures. Use of thermoplastic stabilization has received attention for waste containing both hazardous and radioactive wastes (Swindlehurst et al; 1989). In an effort to reuse of contaminated materials, soils contaminated with mineral oils were stabilized with bitumen and added to road paving materials (Fall et al; 1997).

2.2.4 Tests for Evaluating Effectiveness of S/S Treatment

The degree of effectiveness of S/S treatment requires the measurement of physical, engineering and chemical properties of the stabilized/solidified material. Malviya and Chaudhary (2006) reported that the degree of effectiveness of S/S treatment can be defined basically by two parameters, the strength and the leach resistance of the treated product. In the study on evaluation of effectiveness of S/S treatment, LaGrega et al., 2001 included the micro-structural examination of the stabilized/solidified mass also which makes better understanding of the nature of the S/S process. X-ray diffraction (XRD)/Scanning Electron Microscope (SEM) tests are used for the micro-structural examination (Means et al. 1995; Qian et al 2006). Hills and Pollard (1997) used setting and strength development as indicators of solidification and leach test to assess the extent of fixation (immobilization). Yin et al (2007) used strength development and leachability to determine the effectiveness of S/S technology for treating steel scrap yard contaminated with heavy metals, using ordinary Portland cement as the binder. Some of the tests that are normally carried out to evaluate the effectiveness of the S/S technology include the following

- Compressive strength test
- Permeability tests

- Leachability using toxicity characteristics leaching procedure (TCLP) tests

2.2.4.1 Compressive strength test

Strength testing is often used during a treatability study to indicate how well a material will endure mechanical stresses created by overburden and earth moving equipment (USACE, 1995). Strength test data often provide a baseline comparison between stabilized and unstabilized waste materials. Unstabilized contaminated soil generally does not exhibit good strength, but with cement stabilization, the strength is expected to increase significantly.

The unconfined compressive strength (UCS) test is the recommended test used in testing the strength of S/S treated soil. This test is usually accomplished with help of unconfined compression machine and varying maximum load. UCS test is performed according to ASTM D2166-85. UCS test is usually performed at different time intervals of 1, 3, 7, 14, 28, 90, etc days to monitor the effect of the changes in the mineralogical composition of treated product, with increasing time and environmental exposure. Environment Protection Agency (EPA) of USA has specified the minimum required unconfined compressive strength for a stabilized/solidified material to be 0.35 MPa (50 psi). However, in UK, acceptable 28-day unconfined compressive strength is 0.7 MPa (100 psi). The requirements of UCS may be increased depending on the utilization of treated products. Yin et al (2007) have reported that for using stabilized waste as mortar the minimum unconfined compressive strength should be 20 MPa (2900 psi). A study by Stegemann et al. (1990) reported unconfined compressive strength in the range of 0.06 to 19.99 MPa (9 to 2900 psi) for 69 stabilized/solidified wastes.

2.2.4.2 Permeability test

Permeability is the ability of a material to conduct or discharge water in response to an applied hydraulic gradient. This parameter is important in that it indicates the ease with which water will pass through the stabilized/solidified material (Poon et al, 1986). It gives information about the rate at which leachates in the treated waste might migrate into the environment. Permeability test is not a measure of leachability because having higher permeability does not infer that the waste has not been treated well. Factors affecting the permeability include density, degree of saturation, type of permeating fluid, hydraulic gradient and particle size distribution. For stabilization/solidification to be effective, the permeability of treated product should be in the range of 10^{-6} to 10^{-10} m/s (USEPA, 1989). For stabilized/solidified products to be utilized for land burial, permeability should be less than 10^{-7} m/s was.

The triaxial permeability test has been recommended for evaluating effectiveness of stabilization/solidification in reducing the permeability. Consideration must be given to the confining pressure, gradient and the permeating fluids which will reflect the field condition. Yilmaz et al (2003) working on solidification/stabilization of hazardous wastes containing metals and organic contaminants used Portland cement as the only reagents for stabilization obtained a range of 1.04×10^{-9} to 2.1×10^{-9} m/s as the permeability values.

2.2.4.3 Leachability test

The effective performance of stabilized wastes is measured in terms of leaching and extraction tests. Several leaching tests are employed to evaluate the effectiveness S/S

process in reducing the leachability. Toxicity characteristics leaching procedure (TCLP) test; synthetic acid precipitation leach procedure (SPLP) test; American nuclear society leach test (ANSI/ANS/16.1); dynamic leach test etc are among the leaching tests. TCLP test method is commonly used by the EPA to determine if a waste is still hazardous or otherwise (Qian et al., 2006). TCLP is designed to simulate the leaching potential of a waste within an unmanaged landfill designed for municipal refuse. TCLP leaching solution (acetic acid) is designed to simulate worst-case leaching conditions on disintegrated landfill wastes due to aging effects, while the SPLP uses a leaching solution (nitric/sulphuric acid) that simulates acid rain.

For the TCLP test, samples are crushed and are made to pass through 9.5 mm sieve, simulating a long-term effect of stabilized/solidified material which might breakdown due to overburden stresses. The pulverized samples is mixed with an acetic acid solution (pH = 2.88 ± 0.05 if solid pH is higher than 5 or pH= 4.93 ± 0.05 if solid pH is lower than 5) at a solution/solid ratio of 20. The suspension is then tumbled for 18 hours in a rotary extractor at 30 ± 2 rpm and following this, separation of the extract solution from the solids is achieved by vacuum filtration (LaGrega et al. 2001). Soluble contaminants concentrations in the solution are measured using an inductively coupled plasma-optical emission spectrometer (ICP-OES) or the atomic absorption spectrometer (AAS). The TCLP extracts is analyzed for hazardous waste constituents. Table 2.4 shows the EPA regulatory limit to check if the waste is still hazardous or not.

Table 2.4 : Maximum concentration of metal contaminants for TCLP (USEPA, 1999)

Heavy Metals	HW Number	EPA Method	Regulatory Limit
Arsenic	D004	7061/6010	5.0 mg/l
Barium	D005	7081/6010	100 mg/l
Cadmium	D006	7131/6010	1.0 mg/l
Chromium	D007	7191/6010	5.0 mg/l
Lead	D008	7421/6010	5.0 mg/l
Mercury	D009	7470/6010	0.2 mg/l
Selenium	D110	7741/6010	1.0 mg/l
Silver	D011	7761/6010	5.0 mg/l

In determining the leaching characteristics of S/S material, Yin et al. (2007) used TCLP, SPLP and the whole block leaching test. The whole block leaching was designed to simulate short-term leaching conditions of intact monolithic products. The results obtained on stabilizing/solidifying of a metal scrap yard conform to the regulatory standards. Hexavalent chromium concentrations in solution were evaluated using a UV–Vis spectrophotometer (Dermatas and Meng, 2003). All TCLP testing was performed on sample duplicates and average values were used. In addition, all analyses were performed by using two different quality control standards, as well as the method of standard additions (spiking), to ensure proper quality control of the reported results (Dermatas and Meng, 2003). After homogenization, the suspension was allowed to stand for 7 days, and soluble contaminants concentration will be determined according to the standard methods.

2.3 TREATMENT OF CONTAMINATED SOILS USING STABILIZATION/SOLIDIFICATION (S/S) TECHNOLOGY

Dermatas and Meng (2003) conducted study on S/S treatment of heavy metal contaminated soils using fly ash blended quicklime as binder. They used two types of heavy metals (lead and hexavalent chromium) to artificially contaminate the clayey-sand

soils. The different levels of lead and chromium used for contaminating the soil are given in Table 2.5. They evaluated effectiveness of the S/S treatment by measuring unconfined compressive strength, swell, and TCLP of the treated soils. The S/S treatment was found to be effective in reducing heavy metal leachability well below the permissible limits. Stress-strain properties of the treated soils were also improved significantly thus allowing their reuse as readily available construction materials. However, a problem of swelling due to formation of ettringite in the presence of sulfate was detected in treating the soils using quicklime-fly ash binder.

Table 2.5 : Heavy metal contents in artificially contaminated soils (Dermatas and Meng, 2003)

Contaminant Source		**Heavy metal species soil Concentration (mg/kg soil)**
$Cr(NO_3)_3$	Chromium Nitrate	4000
K_2CrO_4	Potassium Chromate	4000
PbO	Lead Oxide	7000

Yin et al. (2006) reported a study on S/S treatment of lead-contaminated soil using ordinary Portland cement (OPC) and rice husk ash (RHA) as binder. The contaminated different samples of soil by varying lead concentrations (in the form of nitrates). They evaluated effectiveness of the treatment by measuring physical properties (unconfined compressive strength, setting times, etc) and chemical properties (leachability of lead, pH, alkalinity of leachability) of the treated soil samples. Test results indicated that usage of OPC admixed with RHA as an overall binder system for S/S of lead-contaminated soils is more favorable in reducing the leachability of lead from the treated samples than a binder system with OPC alone. However, partial replacement of OPC with RHA in the binder system reduced the unconfined compressive strength of solidified samples.

Yin et al. (2007) used OPC as the binder in the cleaning of soil contaminated metal scrap yard. For characterizing the soil for heavy metals concentrations, the soil was acid digested and the chemical analysis carried out using the OPTIMA 3000 Perkin-Elmer ICP-OES. Test results revealed that the soil contains heavy metals that exceeded the limit of EPA making the soil hazardous. Table 2.5 shows the heavy metal concentration in the soil after analysis. After characterization, three levels of binder/waste ratio viz 0.5, 1 and 2 were used for S/S treatment. The effectiveness of the S/S treatment was evaluated in terms of strength and leachability of the treated soil. The minimum strength criteria was met in all three cases, however, the leachability was found within the permissible limit only in two cases of binder/waste ratios of 1 and 2. The binder/waste ratio of 1 was found to be the most appropriate for stabilizing the soil to limit specified by the EPA.

Table 2.6 : Heavy metal concentrations in contaminated soil (Yin et al. 2007)

Heavy metal	**Concentration (mg/kg)**
Iron Fe	42,194
Chromium Cr	52
Cadmium Cd	ND
Zinc Zn	690
Lead Pb	428
Copper Cu	107
Aluminium Al	8874

ND denotes ‘below detection limits’.

Moon et al. (2008) have reported a study on assessment of the performance of cement kiln dust (CKD) as a binder for S/S of arsenic contaminated soils. Laboratory-prepared slurries, made of either kaolinite or montmorillonite, and field soils spiked with either As^{3+} or As^{5+} were prepared and treated with CKD ranging from 10 to 25% (by mass). Sodium arsenite and sodium arsenate at 0.1 % (by mass) were used to simulate arsenite (As^{3+}) and arsenate (As^{5+}) source contamination in soils, respectively. The effectiveness

of treatment was evaluated at curing periods of 1- and 7-days based on the TCLP. Arsenic–CKD and Arsenic–clay–CKD slurries were also spiked at 10 wt% to evaluate Arsenic immobilization mechanism using X-ray powder diffraction (XRPD) analyses. Overall, the TCLP results showed that only the As^{5+} concentrations in kaolinite amended with 25 % (by mass) CKD after 1 day of curing were less than the TCLP regulatory limit of 5 mg/L. Moreover, at 7 days of curing, all As^{3+} and As^{5+} concentrations obtained from kaolinite soils were less than the TCLP criteria. However, none of the CKD-amended montmorillonite samples satisfied the TCLP–Arsenic criteria at 7 days. Only field soil samples amended with 20 % (by mass) CKD complied with the TCLP criteria within 1 day of curing, where the source contamination was As^{5+}. XRPD and scanning electron microscopy (SEM)–energy dispersive X-ray spectroscopy (EDX) results showed that Ca–As–O and NaCaAsO4·7.5H2O were the primary phases responsible for As^{3+} and As5+ immobilization in the soils, respectively.

Moon et al. (2009) conducted a research work on S/S treatment of selenium contaminated soil using Portland cement and cement kiln dust as a binder. S/S processes were used to immobilize selenium (Se) as selenite (SeO_3^{2-}) and selenate (SeO_4^{2-}). Artificially contaminated soils were prepared by individually spiking kaolinite, montmorillonite and dredged material (DM; an organic silt) with 1000 mg/kg of each selenium compound. After mellowing for 7 days, the Se-impacted soils were each stabilized with 5, 10 and 15% Type I/II Portland cement (P) and cement kiln dust (C) and then were cured for 7 and 28 days. The TCLP was used to evaluate the effectiveness of the S/S treatments. At 28 days curing, P doses of 10 and 15% produced five out of six TCLP-Se(IV) concentrations below 10 mg/L, whereas only the 15% C in DM had a

TCLP-Se(IV) concentration less than 10 mg/L. Several treatments satisfied the EPA TCLP best demonstrated available technology (BDAT) limits (5.7 mg/L) for selenium at pozzolan doses up to 10 times less than the treatments that established the BDAT. Neither pozzolan was capable of reducing the TCLP-Se(VI) concentrations below 25 mg/L. Se-soil–cement slurries aged for 30 days enabled the identification of Se precipitates by X-ray powder diffraction (XRD) and scanning electron microscopy (SEM)–energy dispersive X-ray spectroscopy (EDX). XRD and SEM–EDX analyses of the Se(IV)- and Se(VI)-soil–cement slurries revealed that the key selenium bearing phases for all three soil–cement slurries were calcium selenite hydrate ($CaSeO^3 \cdot H_2O$) and selenate substituted ettringite ($Ca_6Al_2(SeO4)_3(OH)_{12} \cdot 26H_2O$), respectively.

Choi et al. (2009) reported that the technology was used in the cleanup of 1800 cubic yard of lead-contaminated soil. Cement was used as the binder.

CHAPTER THREE

PRELIMINARY EXPERIMENTAL INVESTIGATION

3.1 INTRODUCTION

Before conducting a detailed experimental investigation to achieve the objectives of the present study, a preliminary experimental investigation was carried out for the following purposes:

- Selection and characterization of two types of local soils
- Selection of two types of industrial hazardous wastes and their dosages for contaminating the soils for S/S treatment
- Selection of the binders for S/S treatment under this study
- Selection of a suitable size of the test specimens used for evaluating effectiveness of the treated soils
- Selection of a suitable range of moisture content for mixing contaminated soil and binder
- Selection of optimum moisture content and optimum dosage of FFA for both types of selected soils

This chapter deals with the details of the tests conducted, test results obtained and data interpretation for the above purposes.

3.2 SELECTION AND CHARACTERIZATION OF TWO TYPES OF LOCAL SOILS

Two types of soil (marl and sand), prevalent in eastern Saudi-Arabia, were selected for this study. For characterization of soils and later on using for detailed investigation, marl soil was collected from the construction site around the stadium of the KFUPM while the sand soil was brought from outside KFUPM. The soil samples were sieved to remove the large boulders samples retained on the No. 4 sieve) and oven-dried for 24 hours at 110^{o}C. Specific gravity test, grain size analysis of marl using hydrometer test, plasticity test on marl, sieve analysis of sand were conducted to characterize the selected soils.

3.2.1 Specific Gravity Tests

The soil samples passing sieve No. 4 were used for specific gravity tests in accordance with ASTM D 854. Specific gravities of marl and sand were found to be 2.77 and 2.66, respectively.

3.2.2 Grain Size Distribution

The grain size distribution analysis was conducted on the marl samples using washed sieving techniques as per the ASTM D 422. After observing that the marl soil sample contains a significant amount of the soil particles passing through No 200 ASTM sieve, the hydrometer test was conducted on the marl samples to establish the amount of silt in the marl soil. In case of sand, only the dry technique (i.e., sieving) was used for the grain size distribution analysis. The grain size distribution curves for marl and sand soils are shown in Figures 3.1 and 3.2, respectively.

3.2.3 Plasticity Tests

The plasticity tests for determining liquid limit and plastic limit were performed on the marl soil samples passing through No. 40 ASTM sieve in accordance with ASTM D 4318. It was found that the marl soil is non-plastic indicating that the fines in the marl are silt not clay.

3.2.4 Classification of the Selected Soils

Results of plasticity tests indicate that the marl soil under consideration is non-plastic marl. From the observation of Figure 3.1, the given marl soil can be classified as “silty sand (SM)” according to the USCS classification criteria and as “A-1-b” according to the AASHTO classification criteria.

For classifying the sand soil, the uniformity coefficient and the coefficient of curvature were determined to be 3 and 1.33, respectively. Based on the values of uniformity coefficient and the coefficient of curvature and the particle size distribution curve for sand as shown in Figure 3.2, the sand soil can be classified as “poorly graded sand (SP)” according to the USCS classification criteria and as “A-3” according to the AASHTO classification criteria.

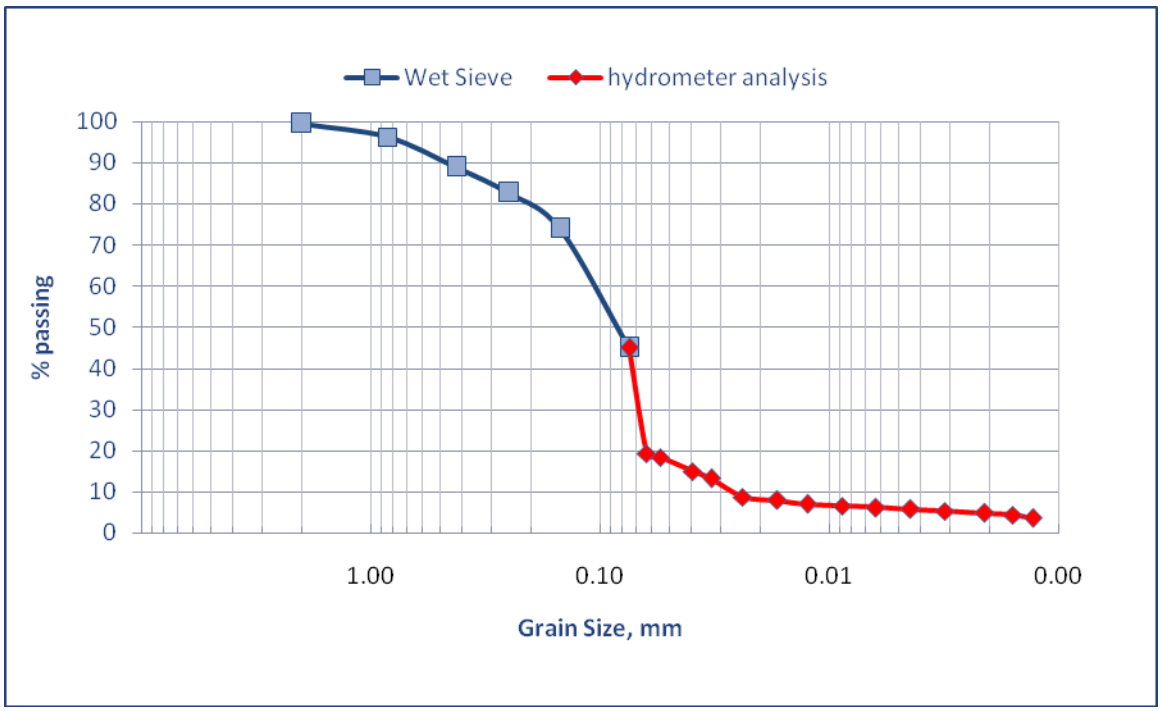

Figure 3.1: Grain size distribution curve for marl

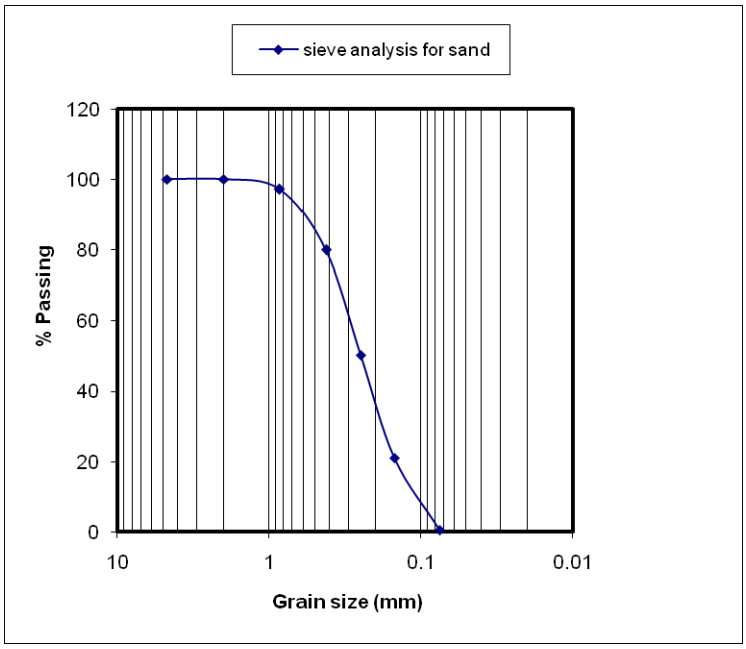

Figure 3.2 : Sieve analysis for sand

Characteristics of the selected soils are summarized in Table 3.1.

Table 3.1: Soil Characteristics

Property	Designation	Sand	Marl
Specific Gravity	ASTM D 854	2.66	2.77
Liquid Limit	ASTM D 4318	-	Non Plastic
Plastic Limit	ASTM D 4318	-	Non Plastic
Classification	USCS	SP	SM
	AASHTO	A-3	A-1-b

3.3 SELECTION OF TYPES AND DOSAGES OF HAZARDOUS WASTES

Steel and electroplating industries' wastes were selected for this study due to their abundance and potential hazardous characteristics because of very high concentrations of some of the heavy metals present in the wastes from these industries.

Dosages for each industrial waste were selected based on the limited information available in literature (Brigden et al. 2000; Asavapisit and Chotklang, 2004) pertaining to the concentration of heavy metals in soils contaminated by wastes from steel and electroplating industries. Typical dosages of heavy metals belonging to both types of hazardous wastes selected for contaminating the soil samples are presented in Table 3.2.

Analytical grades of the compounds containing various heavy metals required for artificially contaminating the soils were used. Calculation of the percentages of heavy

metals in their respective analytical grade compounds is presented in Table 3.3. The percentage of each metal in the respective compound was calculated as follows

$$\%\ \text{of metal in compound} = \frac{\text{Mol. wt. of metal}}{\text{Mol. wt. of compound}} \times \text{purity of compound}$$

Table 3.2: Typical dosages of heavy metals for contaminating the soil samples

Heavy Metals	**Steel** (mg/kg)	**Electroplating** (mg/kg)
Lead (Pb)	20	1
Chromium (Cr)	50	1700
Cadmium (Cd)	2	1
Zinc (Zn)	50	105
Iron (Fe)	8000	200
Copper (Cu)	25	1
Nickel (Ni)	50	-
Manganese (Mn)	500	-
Total	**8697**	**2008**

Table 3.3: Percentage of heavy metals in analytical grade compounds used as source of heavy metals

Analytical grade compound	**Heavy metal**	**% purity of compound**	**Mol. wt. of compund**	**Mol. wt. of heavy metal**	**% of heavy metal in compound**
$Pb(NO_3)_2$	Pb	99.5	331.2	207.2	62.25
$CdCO_3$	Cd	99.7	172.41	112.41	65.00
K_2CrO_4	Cr	99.5	194.19	52.00	26.64
$CuSO_4$	Cu	99	159.61	63.55	39.42
$NiSO_4.6H_2O$	Ni	99	262.85	58.69	22.11
$FeSO_4.7H_2O$	Fe	99.5	278.01	55.85	19.99
$MnSO_4.H_2O$	Mn	99	169.01	54.94	32.18
ZnO	Zn	99.5	81.38	65.39	79.95

Using the heavy metal content of each analytical grade compound, the required dosages of the analytical grade compounds were calculated for each type of heavy metal and waste, as shown in Table 3.4.

Table 3.4: Dosages of analytical grade compounds used for artificially contaminating the soil samples with heavy metal

Heavy metal	Intended dosage of heavy metal (mg/kg of soil)		Analytical grade compound	Dosage of compound (mg/kg of soil)	
	in steel waste	in electroplating waste		in steel waste	in electroplating waste
Pb	20	1	$Pb(NO_3)_2$	32	2
Cd	2	1	$CdCO_3$	3	2
Cr	50	1700	K_2CrO_4	188	6380
Cu	25	1	$CuSO_4$	63	3
Ni	50	--	$NiSO_4.6H_2O$	226	--
Fe	8000	200	$FeSO_4.7H_2O$	40022	1001
Mn	500	--	$MnSO_4.H_2O$	1554	--
Zn	50	105	ZnO	63	131

3.4 SELECTION OF BINDERS FOR S/S TREATMENT

Ordinary Type I Portland cement (OPC) blended with an adequate percentage of fuel fly ash (FFA), locally available from Shoiba plant of Saudi Aramco, was selected as binder for S/S treatment. Table 3.5 shows the elemental compositions of both the OPC and FFA used in this study as determined by the Jeol scanning electron microscope (SEM) model JSM-5800LV to characterize the binders.

Table 3.5 : Elemental composition of OPC and FFA used in the study

Element	OPC		FFA	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
Oxygen (O)	46.18	66.27	29.68	31.66
Carbon (C)	Nil	Nil	32.52	46.20
Magnesium (Mg)	3.11	2.94	19.20	13.48
Aluminium (Al)	0.71	0.60	0.44	0.28
Silicon (Si)	6.94	5.68	0.33	0.20
Sulphur (S)	1.39	1.00	11.42	6.08
Calcium (Ca)	39.45	22.60	0.31	0.13
Iron (Fe)	2.22	0.91	0.50	0.15
Vanadium (V)	Nil	Nil	4.11	1.38
Chromium (Cr)	Nil	Nil	0.08	0.03
Manganese (Mn)	Nil	Nil	0.41	0.13
Nickel (Ni)	Nil	Nil	1.01	0.29

3.5 SELECTION OF A SUITABLE SIZE FOR TEST SPECIMENS

Sizes of the specimens used for unconfined compressive strength test are commonly decided keeping a height/diameter ratio between 2 and 3 with diameters of 38 mm (1.5 inch) or 100 mm (4 inch) (Al-Khafaji and Andersland, 1992). From the convenience point of view, same specimen size (38 mm diameter and 76 mm heigth) was selected for conducting unconfined compressive strength, porosity and permeability tests. A relatively smaller specimen size keeping a height/diameter ratio of 2 was selected considering convenienc in conducting permeability test and also satisfying the range of height/diameter ratio recommended for unconfined compressive strength test. Figure 3.3 shows a photographic view of the moulds fabricated for casting specimens of the selected size.

Figure 3.3 : View of the selected mould size (1.5 inch × 3 inch)

The adequacy of the selected size of the specime was examined through standard Proctor compaction test on the marl soil (as sand soil could not be compacted using the compaction practice used in standard Proctor test) as per ASTM D 698. The weight of hammer and height of drop of hammer for compacting the soil in selected mould were kept as 0.5 kg (1.1 lb) and 101.6 mm (4 inch), respectively. Even the weight hammer and height of drop in case of the selected specimen size were one-fifth and one-third, respectively, of that for the standard specimen size, the compaction energy for the selected specimen size was around 72% of that for the satandard size specimen.

Plots of the moisture-density data obtained through compaction tests conducted on standard size and selected size specimens are shown in Figure 3.4.

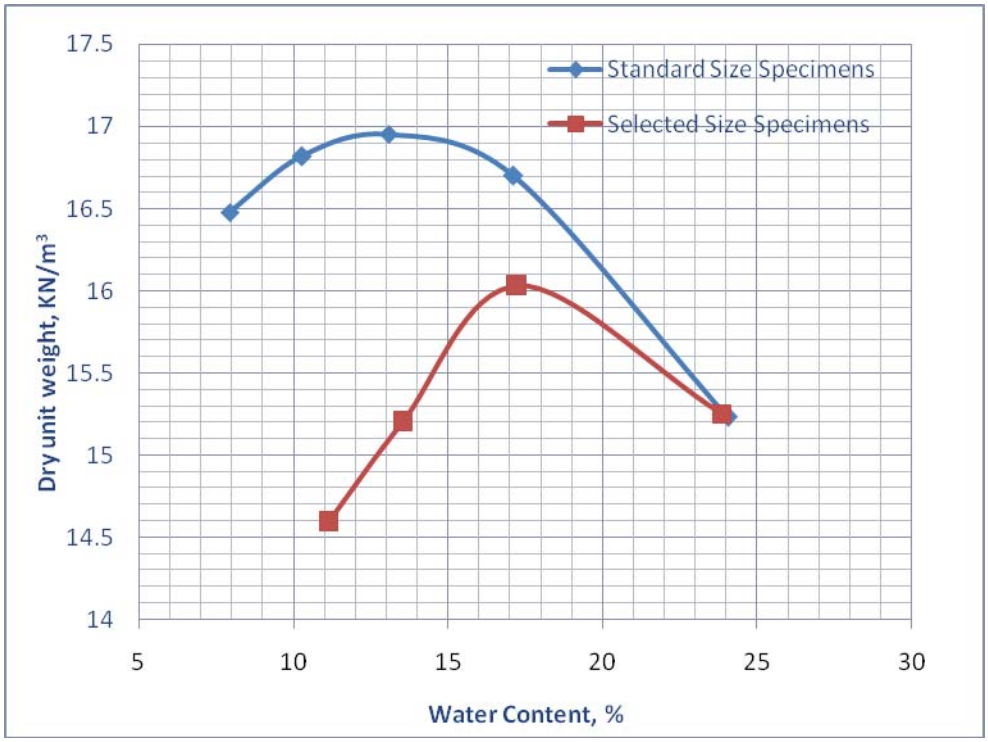

Figure 3.4: Moisture-density curves for standard and selected size specimens

From the observation of the moisture-density curves, as shown in Figure 3.4, the maximum dry unit weights of standard specimen size and selected specimen size were found to be 16.9 kN/m^3 (108 lb/ft^3) and 16 kN/m^3 (102 lb/ft^3) corresponding to optimum moisture contents of 13% and 17%, respectively. The selected specimen size has a maximum unit weight of about 95% of the maximum unit weight for the standard specimen size. Therefore, the selected specimen size is found to be adequate. A higher optimum moisture content in case of selected specimen size is due to comparatively low compaction energy.

The summary of the compaction tests for examining adequacy of the selected specimen size is presented in Table 3.6.

Table 3.6: Summary of compaction tests

Compaction test parameters	Standard size (101.6 mm × 116.3 mm)	Selected size (38.1 mm× 76.2 mm)
Number of layers	3	3
Weight of hammer	2.5 kg	0.5 kg
Height of drop of hammer	304.8 mm	101.6 mm
Number of blows	25	25
Compaction energy per unit volume	592.5 kj/m^2	424.9 kj/m^2
Maximum dry unit weight	16.9 kN/m^3	16 kN/m^3
Optimum moisture content	13 %	17 %

3.6 SELECTION OF SUITABLE RANGE OF MOISTURE CONTENTS

For determining a suitable range of moisture content which can be used for mixing the contaminated soil with binder for S/S treatment, four mixtures of marl soil and OPC (soil alone; soil and OPC in 10 to 1 ratio by mass; soil and OPC in 4 to 1 ratio by mass; soil and OPC in 2 to 1 ratio by mass) were prepared at different trial moisture contents for conducting the compaction tests using the selected mould (1.5 inch × 3 inch). The results of the compaction tests were plotted as shown Figure 3.5

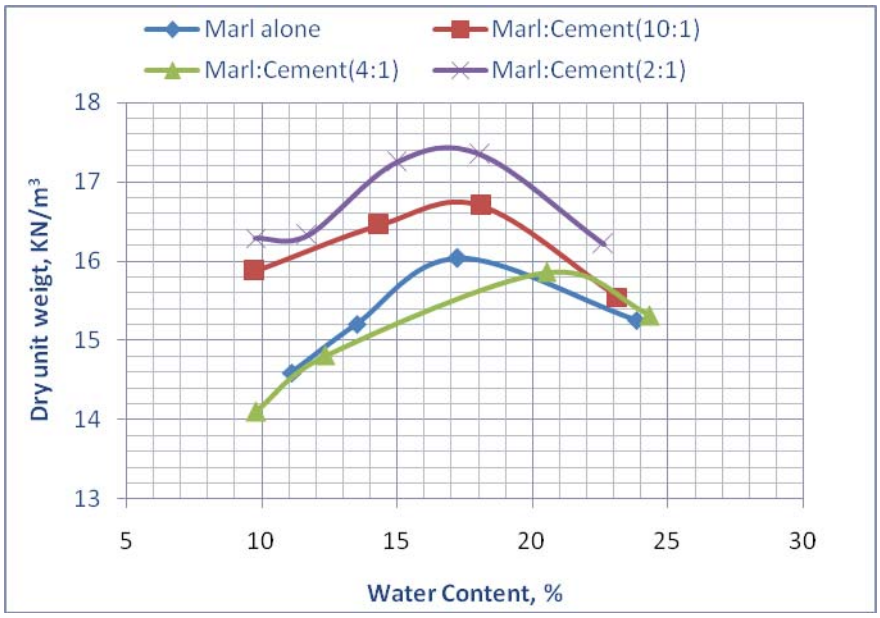

Figure 3.5 : Compaction curves for the four mixtures of marl and OPC

From Figure 3.5, it is observed that the optimum moisture contents of the mixtures of marl soil and OPC vary between 17% and 21%. According to a study conducted at KFUPM by Qahwash (1989), the optimum moisture contents of the mixtures of the local sand (found in Dammam-Dhahran region) and a cementitious material (calacreous sediment) were found to vary between 10% and 14%.

Therefore, the ranges of moisture contents for marl and sand were consider as 17 to 21% and 10 to 14%, respectively, for the purpose of selecting a suitable moisture content and FFA content for each type of soil based on the results of unconfined compressive strength tests.

3.7 SELECTION OF OPTIMUM MOISTURE CONTENT AND OPTIMUM DOSAGE OF FFA

For selecting optimium moisture content and optimum dosage of FFA to be used for S/S treatment of both types of soil under the present study, 15 mixtures of soil and binder (OPC + FFA) were prepared by varying moisture content in suitable ranges (i.e., 17 to 21% for marl and 10 to 14% for sand) and varying the FFA content in the range of 5 to 20%. For each mixture, the soil to binder ratio was kept constant at 2.5 to 1. These soil-binder mixtures were cast in the mould of selected size and tested for unconfined compressive strength (UCS) after 7 days air-curing. Based on the results of the UCS tests, optimum values of moisture content and dosage of FFA were selected for both types of soil.

3.7.1 Selection of Optimum Moisture Content and Optimum Dosage of FFA for Marl Soil

The UCS test results obtained for marl soil-binder mixtures are presented in Table 3.7. For the purpose of selecting an optimal FFA and moisture content for marl soil, the UCS test results are plotted as shown in Figure 3.6.

Table 3.7 : UCS test results for marl soil-binder mixtures

Soil-Binder Mixture	Average 7-day UCS (kPa)		
	17% water	19% water	21% water
100%Cement	8084.1	6236.3	5602.2
95%Cement + 5%FFA	5143.5	2023.6	2302.8
90%Cement + 10%FFA	2196.0	2585.5	1934.0
85%Cement + 15%FFA	1072.1	1303.1	1668.5
80%Cement + 20%FFA	599.8	1082.5	1261.7

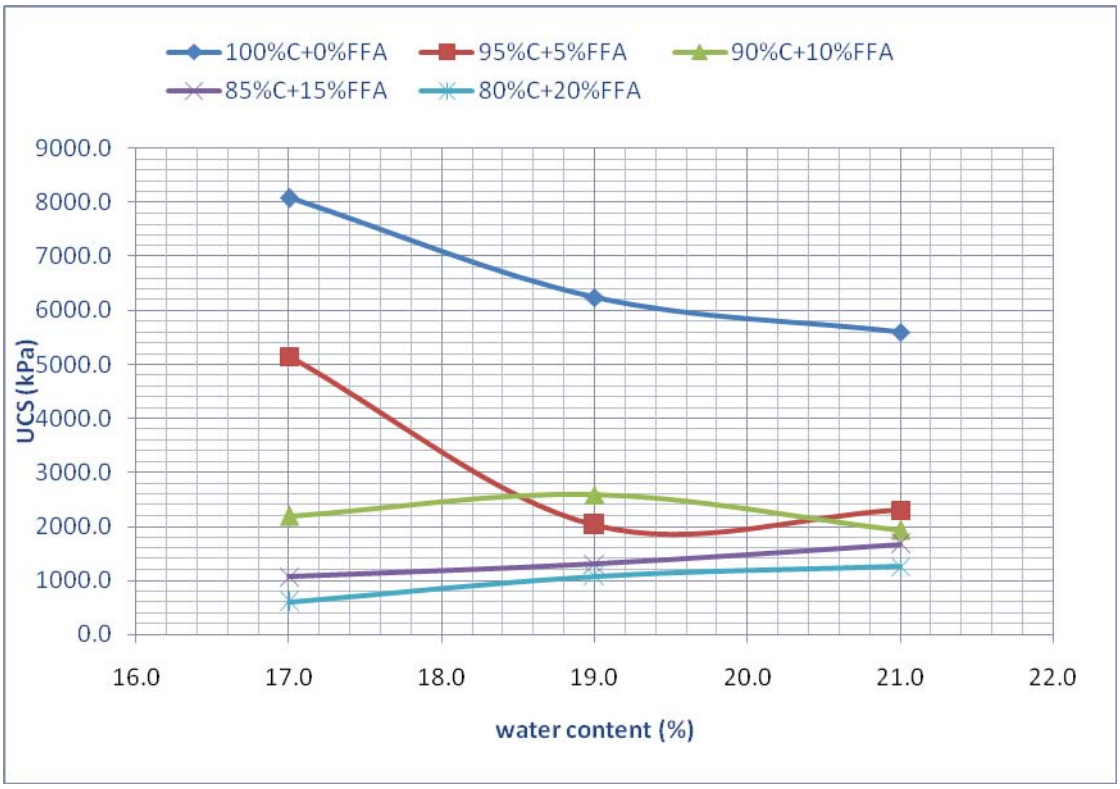

Figure 3.6 : Plots of UCS test results of marl-binder mixtures

From Figure 3.6, it is observed that at 17% moisture content and 5% FFA content the UCS is the highest among all cases of FFA incorporation. However, 5% of FFA is of negligible amount and would not significantly reduce the cost of the binder. Therefore, optimum FFA content and moisture content for marl soil was selected to be 10% and 19%, respectively.

3.7.2 Selection of Optimum Moisture Content and Optimum Dosage of FFA for Sand Soil

The UCS test results obtained for sand soil-binder mixtures are presented in Table 3.8. For the purpose of selecting an optimal FFA and moisture content for sand soil, the UCS test results are plotted as shown in Figure 3.7.

Table 3.8 : UCS test results for sand soil-binder mixtures

Soil-Binder Mixture	Average 7-day UCS (kPa)		
	10% water	12% water	14% water
100%Cement	2309.7	6205.3	3233.6
95%Cement + 5%FFA	1234.2	2564.8	2068.4
90%Cement + 10%FFA	1034.2	1937.4	1758.2
85%Cement + 15%FFA	848.1	1475.5	827.4
80%Cement + 20%FFA	579.2	1103.2	510.2

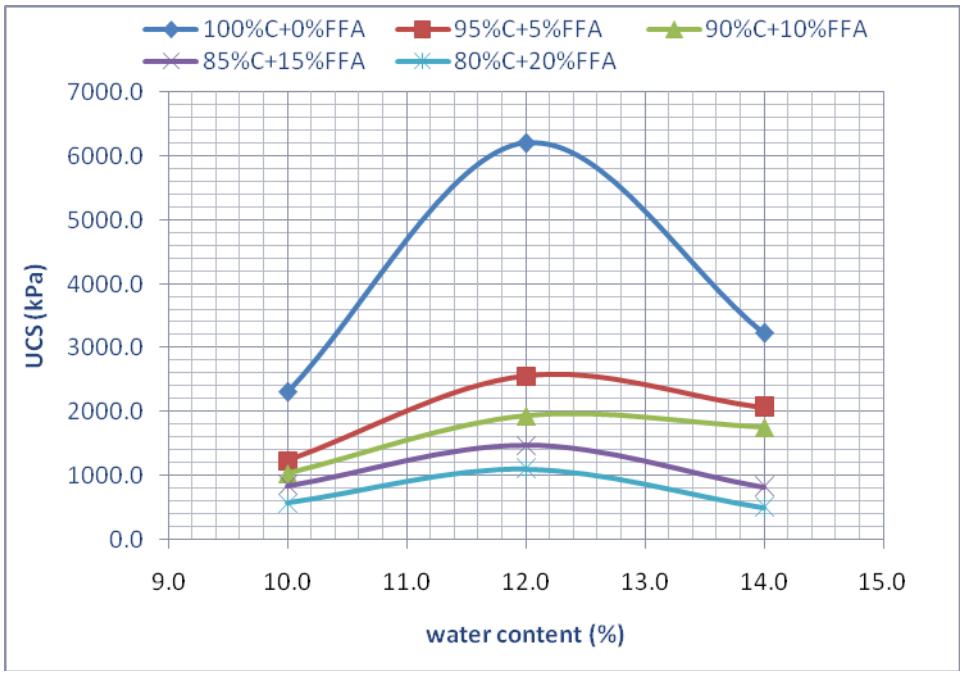

Figure 3.7 : Plots of UCS test results of sand-binder mixtures

From Figure 3.7, it is observed that at 12% moisture content and 5% FFA content the UCS is closest to the value corresponding to the 100% cement case. However, 5% of FFA is of negligible amount and would not significantly reduce the cost of the binder. Therefore, optimum FFA content and moisture content for sand soil was selected to be 10% and 12%, respectively.

3.8 SUMMARY

The outcomes of the preliminary experimental investigation are summarized as follows:

- Selected two types of soils are classified as follows:
  - Marl as: non-plastic silty sand (SM)
  - Sand as: poorly-graded sand (SP)
- Dosages of analytical grades of compounds for inducting heavy metals in intended concentrations (Table 3.4)
- Size of specimen for conducting UCS, porosity and permeability is selected as 1.5 inch × 3.0 inch
- Optimum moisture contents were selected as 19% and 12% for marl and sand, respectively
- Optimum dosage of FFA was selected as 10% for both types of soils

CHAPTER FOUR

DETAILED EXPERIMENTAL PROGRAM: METHODOLOGY

4.1 INTRODUCTION

Based on the outcomes of the preliminary experimental investigation, as described in Chapter 3, a detailed experimental program was planned to study the effectiveness of the S/S technique for treating two types of selected soils contaminated with two types of hazardous wastes. Calculated dosages of analytical grades of compounds for inducting heavy metals in intended concentrations; selected size of specimen for conducting UCS, porosity and permeability tests; optimum moisture contents selected for marl and sand; and optimum dosage of FFA selected for both types of soils were used in the detailed experimental program.

Following experimental variables were used in the experiment design:

- Types of soil (two, marl and sand)
- Soil contaminants (three, no contaminant, electroplating waste, and steel waste)
- Binder options (two, OPC alone and OPC blended with FFA)
- Percentages of binder (four, 10%, 20%, 30%, and 40% by mass of contaminated dry soil)

Considering the above experimental variables a total of 48 mixtures were prepared and tested to determine the following:

- Density

- Unconfined compressive strength (UCS)
- Porosity
- Permeability
- Leachability using toxicity characteristics leaching procedure (TCLP)

The flow chart for the experimental program is shown in Figure 4.1

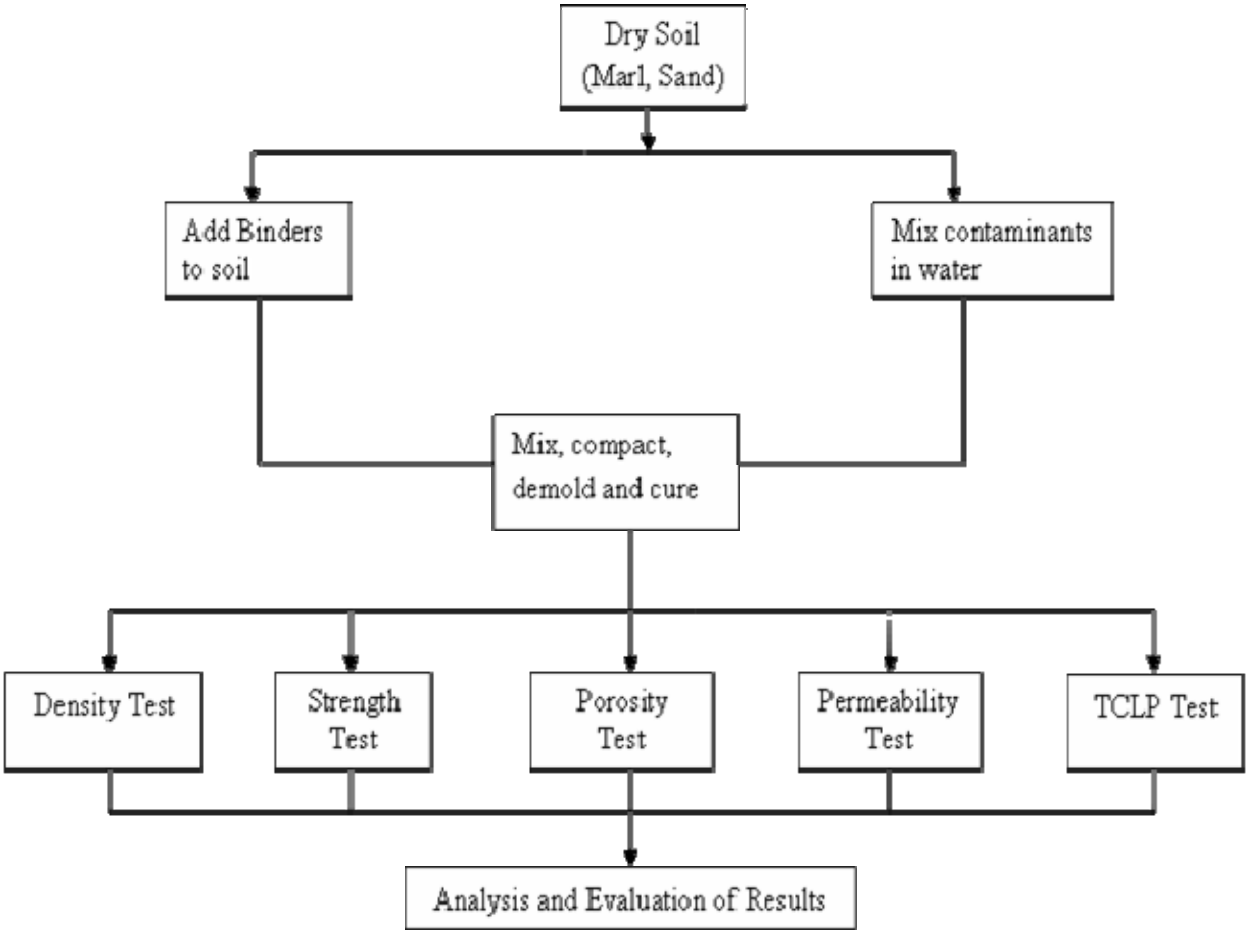

Figure 4.1: Flow chart showing the detailed experimental program

4.2 MIXTURE PROPORTIONS

The mixture proportions worked out considering the experimental variables and their levels are given in Table 4.1 through 4.6. For mixing, the optimum water contents considered

were 19 % and 12 % for marl and sand soil samples, respectively. FFA content of 10% was considered for replacing OPC for both types of soil. These values of water contents and FFA content were optimally selected through the preliminary experimental investigation. A total of 48 mixtures were cast in triplicate.

Table 4.1: Mixture of uncontaminated marl soil and binders (control)

Mix #	Marl (%)	OPC (%)	FFA (%)	Water (%)
1	100	10	-	19
2	100	20	-	19
3	100	30	-	19
4	100	40	-	19
5	100	9	1	19
6	100	18	2	19
7	100	27	3	19
8	100	36	4	19

Table 4.2 : Mixtures of the marl soil contaminated with electroplating waste and binders

Mix #	Marl (%)	Electr. Waste (%)	OPC (%)	FFA (%)	Water (%)
1	99.25	0.75	10	-	19
2	99.25	0.75	20	-	19
3	99.25	0.75	30	-	19
4	99.25	0.75	40	-	19
5	99.25	0.75	9	1	19
6	99.25	0.75	18	2	19
7	99.25	0.75	27	3	19
8	99.25	0.75	36	4	19

Table 4.3 : Mixtures of the marl soil contaminated with steel waste and binders

Mix #	Marl (%)	Steel Waste (%)	OPC (%)	FFA (%)	Water (%)
1	95.785	4.215	10	-	19
2	95.785	4.215	20	-	19
3	95.785	4.215	30	-	19
4	95.785	4.215	40	-	19
5	95.785	4.215	9	1	19
6	95.785	4.215	18	2	19
7	95.785	4.215	27	3	19
8	95.785	4.215	36	4	19

Table 4.4 : Mixture of uncontaminated sand and binders (control)

Mix #	Sand (%)	OPC (%)	FFA (%)	Water (%)
1	100	10	-	12
2	100	20	-	12
3	100	30	-	12
4	100	40	-	12
5	100	9	1	12
6	100	18	2	12
7	100	27	3	12
8	100	36	4	12

Table 4.5 : Mixtures of the sand contaminated with electroplating waste and binders

Mix #	Sand (%)	Electr. Waste (%)	OPC (%)	FFA (%)	Water (%)
1	99.25	0.75	10	-	12
2	99.25	0.75	20	-	12
3	99.25	0.75	30	-	12
4	99.25	0.75	40	-	12
5	99.25	0.75	9	1	12
6	99.25	0.75	18	2	12
7	99.25	0.75	27	3	12
8	99.25	0.75	36	4	12

Table 4.6 : Mixtures of the sand contaminated with steel waste and binders

Mix #	Sand (%)	Steel Waste (%)	OPC (%)	FFA (%)	Water (%)
1	95.785	4.215	10	-	12
2	95.785	4.215	20	-	12
3	95.785	4.215	30	-	12
4	95.785	4.215	40	-	12
5	95.785	4.215	9	1	12
6	95.785	4.215	18	2	12
7	95.785	4.215	27	3	12
8	95.785	4.215	36	4	12

4.3 SAMPLE PREPARATION

All laboratory wares for mixing were pre-cleaned before being used. The reagents Type I OPC and a combination of OPC and FFA were added to the soil samples (marl and sand) and were homogenized thoroughly in the mixer for 10 minutes. The pollutants in form of

compounds of each element were weighed as given in Table 3.4 and added to water to be used for mixing so as to ensure equal mixing of all the pollutants with the soil. The solution was then added to the homogenized soil and reagent in the mixer as shown in Figure 4.2 for 10 minutes. After mixing, the samples were compacted in a 3.81cm by 7.62 cm mold in 3 layers with 25 blows per layer. Therefore, samples were carefully removed from the molds and cured for 180 days in a chamber at a controlled temperature of 22 ± 2^{o}C and humidity > 80%.

Figure 4.2: Mixer used for sample preparation

4.4 DENSITY TEST

The density is a measure of the ratio of the weight of the mass to the volume of the mass. The denser the material the likelihood it will be able to be placed in a landfill or to be used as an underlay in road construction so as to be able to hold the load to be placed on it (Malviya and Chaudhary, 2006).

The diameter and the height were measured at three different locations for each sample and the average values of diameter and height were considered for calculating the volume of the samples. The weights of samples were measured to calculate the density as ratio of the weight of the sample to the volume of the sample.

4.5 UNCONFINED COMPRESSIVE STRENGTH (UCS) TEST

The UCS test is used to determine the undrained strength and stress-strain characteristics of undisturbed, remolded and/or compacted samples. This test is applicable for soils possessing some cohesion, where the sample is not allowed to expel water during loading. The soil sample must retain intrinsic strength after removing the confinement, which is provided by the mold walls for compacted samples. For stabilization/solidification, the UCS is used as a design criterion for placement of samples in landfill or to use samples as sub-base layers. The U.S. EPA specified a minimum value of UCS as 350 kPa for stabilized/solidified materials (Malviya and Chaudhary, 2006).

In this research program, the UCS was performed on samples removed from cylindrical mold of height 3 in. (76.2 mm) and diameter of 1.5 in. (38.1 mm) giving a height to diameter

(h/d) ratio of 2 as recommended by ASTM D 2166. The mold used is the split type; this is to ensure that the samples extruded are as perfect as possible and also ensuring samples' edges are perfect. The test was carried out using the compression machine. A data logger was connected to record the load and deformation. The rate of loading was 1.4 mm/min. The photograph showing the UCS testing is shown in Figure 4.3.

Figure 4.3 : Photograph showing the UCS testing

4.6 POROSITY TEST

The porosity is a measure of the availability of the void spaces in a solid to retain liquids or not (LaGrega et al, 2001). The lesser the pore spaces within the stabilized/solidified materials, the less will be the ability of the material to retain liquid which might cause the disintegration of the material after a long time. The porosity measurement carried out in this research program was porosity by saturation. The procedure followed is as given in the Core Laboratories Incorporated Manuals. The apparatus used for the porosity test is shown in Figure 4.4.

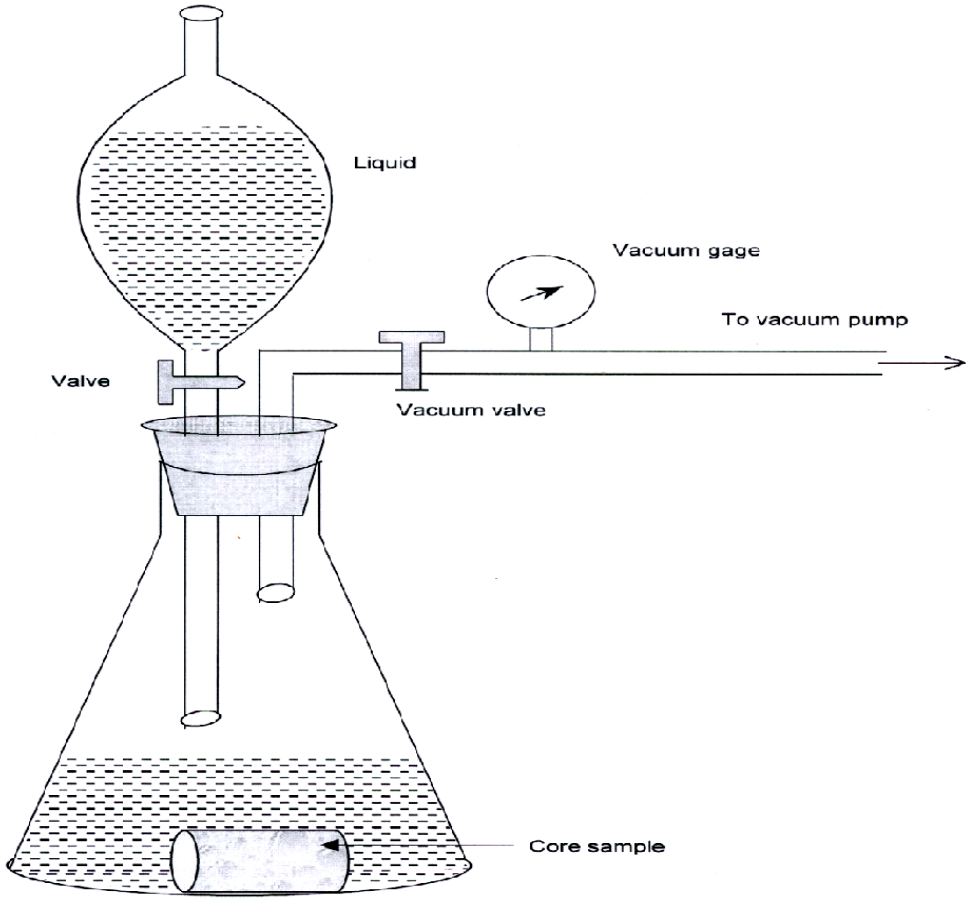

Figure 4.4 : Schematic diagram of the liquid saturation apparatus for determining the porosity

Samples were weighed and the average height and diameter of each sample were recorded. The container was cleaned and dried properly. The samples were arranged properly in the container to save the quantity of liquid to be used for saturating the samples. The liquid used for saturating the samples was a brine solution prepared by dissolving 50 grams of NaCl salt in 1000 ml of deionized water to make a concentration of 50,000 ppm.

The valve leading to the vacuum pump is opened to evacuate all the air within the jar and the pump is kept working for about 4 hours. The samples were then saturated by closing the valve leading to the vacuum pump and opening the valve from the fluid, saturation is continued until the fluid covers the samples. After complete saturation, this valve is now closed and the valve leading to the vacuum pump is opened and the vacuum pump restarted for 30 minutes. This valve is closed again and the other valve is opened to remove the hose to release the air. When there is no vacuum inside the container, the lid opens easily. Samples are taken and the wet weight recorded for calculations of porosity.

Porosity was calculated as follows:

$$\text{Porosity (\%)} = \frac{\text{Pore volume}}{\text{Bulk volume}} \times 100$$

$$\text{Pore volume} = \frac{\text{Weight of fluid}}{\text{Density of brine solution}}$$

$$\text{Weight of fluid} = \text{ Saturated weight of sample } - \text{ Dry weight of sample}$$

$$\text{Bulk volume} = \frac{\pi}{4} D^2 h$$

$D =$ diameter of sample
$h =$ height of sample

4.7 PERMEABILITY TEST

Permeability is a measure of the rate of flow of a fluid through the tortuous pore structure of a material. The permeability of stabilized/solidified material is an important parameter for evaluating the effectiveness of the S/S treatment in reducing leaching of contaminants to the groundwater. The permeability test for this research was carried out using the hydrostatic core-holder assembly. Figure 4.5 shows the experimental set up used for conducting the permeability of the samples after S/S treatment. The procedure followed is as given in the Core Laboratories Incorporated Manuals

The samples used for porosity test were used for permeability test. Sample is inserted into the core-holder rubber sleeve and the sleeve in turn placed inside the core-holder barrel, the upper and lower core-holder caps are tightened and the upper end is connected to the vacuum pump. A positive displacement pump is connected to a transfer cell that has been filled with brine and this is connected to the core-holder inlet. A plastic jar connected to a hand pump which in turn connected to the core-holder annulus is used in creating an overburden pressure around the sample; this help in holding the sample in direct contact with the rubber sleeve and prevent the fluid from flowing in the clearance between the sample and the sleeve. The pump is set up to a flow-rate of 5 ml/s so that the brine in the transfer cell can fully saturate the sample. Increase in flow-rate is applied depending on samples (some samples that are tightly packed require higher flow-rate for the brine to flow through the core). Saturation is allowed for half an hour. After saturation, the pump flow-rate is set at a suitable value, this pushes the brine through the sample and 10 ml of brine is collected at a certain time. The displacing pressure, the volume collected and the time taken to collect the volume are all recorded. The steps are repeated by increasing the flow-rates and recording the corresponding

displacing pressure and time to collect 10 ml of fluid. These values are used in calculating the permeability of the samples.

Permeability of the treated samples was calculated as follows:

$$K = \frac{\gamma}{\mu} K_l$$

where

K is absolute permeability m/s

γ is specific gravity of fluid (brine)

μ is viscosity of fluid (brine) in centipoise

K_l is intrinsic permeability in Darcy(1 Darcy = $9.869233 \times 10^{-13} m^2$)

K_l is determined as:

$$K_l = \frac{1000.m.\mu.L}{A}$$

where: L is length of sample; A is cross-sectional area of sample; and m is the slope of the linearly fitted line between discharge and applied pressure.

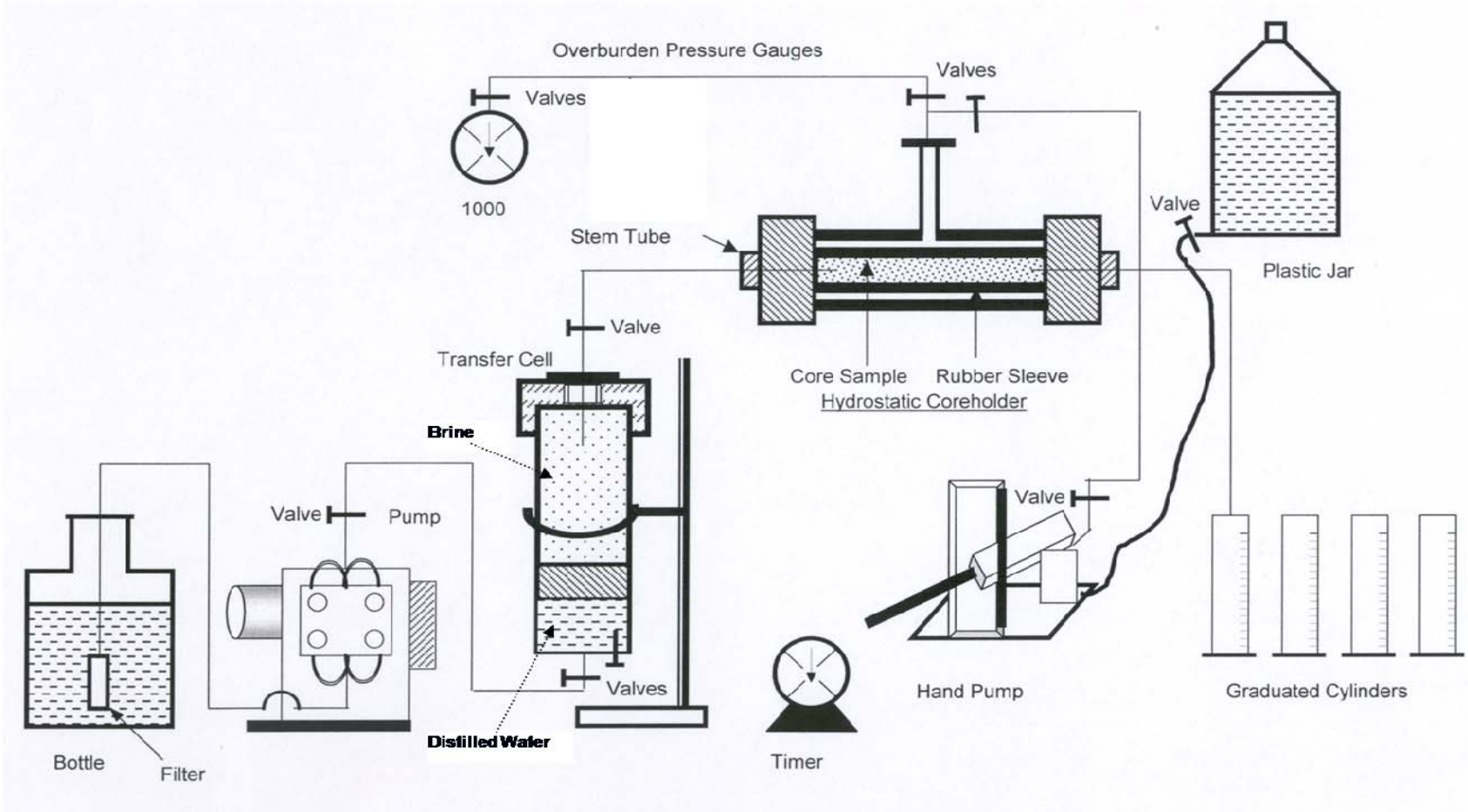

Figure 4.5 : Permeability set-up

4.8 TOXICITY CHARACTERISTICS LEACHING PROCEDURE (TCLP)

The toxicity characteristics leaching procedure (TCLP) method evaluates metal mobility in a landfill (LaGrega et al, 2001). The test simulates worst-case scenario where hazardous waste is co-disposed with municipal waste. The test was carried out in accordance with the USEPA Method 1311. Samples to be tested for the TCLP were selected based on the results from the UCS and permeability tests. Samples were crushed and were made to pass through a standard sieve of 9.5 mm (ASTM sieve 3/8″); this simulates the leaching conditions when treated monolithic wastes ultimately disintegrate subsequent to years of weathering. Samples were then stored in a plastic bag for the extraction (US EPA Method 1311).

4.8.1 Determination of Extraction Fluid

The fluid to be used for the extraction process should either have a pH of 4.93 ± 0.05 or 2.88 ± 0.05; this will depend on the pH of the stabilized/solidified samples that had been crushed. The extracting fluid was selected by taking a sub-sample from each plastic bag (about 5 gram), this was then transferred to a 500 ml beaker, 96.5 ml of deionized water was added and the content was stirred vigorously with a magnetic stirrer on the hot plate for 5 minutes. The pH of all the samples was greater than 5. With the pH greater than 5, 3.5 ml of 1.0N hydrochloric acid was added and the solution was stirred again maintaining the heat at 50^{o}C for 30 seconds. The pH was then measured and was still found to be greater than 5; extraction fluid with pH 2.88 ± 0.05 would be used. The extraction fluid was prepared by diluting 5.7 ml of glacial acetic acid to a volume of 1

litre of deionized in a volumetric flask. The pH was measured after mixing and was found to be 2.85.

4.8.2 TCLP Extraction

All vessels used were precleaned with nitric acid and later rinsed with deionized water. 10 grams of the samples that passed through 9.5 mm sieve were placed in a plastic containers and 200 ml of the extracting fluid was added to provide a ratio of 20:1 mass ratio of leachant to crushed solidified samples. The containers were then agitated using extractor device shown in Figure 4.6 at 30 rpm for 18 hours. After the 18 hours extraction, samples were filtered using the vacuum filtration device shown in Figure 4.7. The device consists of a filter holder (47 mm diameter), a filter cap (300 ml capacity) and filtrate receiver. 0.45 μm membrane filter was used; the filter was cleaned with acid and then rinsed in deionized water. The content from the extraction bottle was slowly poured into the filter funnel and the filtration started. At the completion of the filtration (when no liquid is filtered for 2 minutes), the filtrate was collected in a precleaned polyethylene bottles and this is the TCLP extract (US EPA Method 1311).

4.8.3 Acid Digestion of TCLP Extract

25 ml from the TCLP extract was transferred into a beaker that has been precleaned with acid and deionized water. 3 ml of concentrated nitric acid was added and the beaker was placed on a hot plate. The heating was at a low temperature so as to avoid boiling and there was constant check to prevent drying up of the content since near dryness was needed (2-3 ml). The beaker was allowed to cool, 5 ml of concentrated nitric acid was then added and the beaker was covered with a watch glass for another round of heating

until complete digestion of the acid when there is slight color change. The watch glass is removed and heating is continued to near dryness (2-3 ml). The beaker was cooled and 5 ml of 10% nitric acid was added, this was warmed gently to dissolve any precipitate, then cooled and dilute to volume with deionized water in a 25 ml volumetric flask.

Figure 4.6 : Rotary extractor in operation

Controls (blanks) were prepared for the extraction fluid used as well as for the deionized water so as to ascertain that the results from the samples are quite accurate. Any metal trace found in these blanks will be deducted from the analysis results.

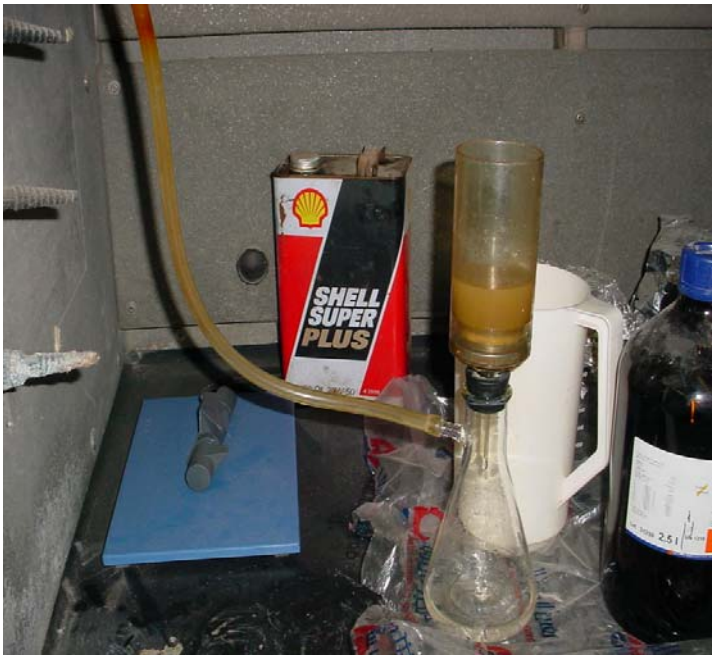

Figure 4.7 : Filtration Device

CHAPTER FIVE

RESULTS AND DISCUSSIONS

All tests for evaluating the effectiveness of S/S treatment under this study were conducted on all the 48 mixtures following the test procedures as described in Chapter 4. In this chapter, all test results are presented and discussions are made based on the data pertaining to the following tests:

- Density
- Unconfined compressive strength (UCS)
- Porosity
- Permeability
- TCLP

5.1 DENSITY

5.1.1 Density of Mixtures of Marl Soil

Test results pertaining to the density of mixtures of marl soil are presented in Table 5.1.

Table 5.1: Density of mixtures of marl soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		Density (g/cm^3)			
M + Cement	180	1.75	1.81	1.85	1.89
M+ E + Cement	180	1.72	1.77	1.78	1.84
M+ Fe + Cement	180	1.63	1.66	1.72	1.78
M + Cement + FFA	180	1.73	1.78	1.80	1.84
M+ E + Cement + FFA	180	1.65	1.68	1.69	1.71
M+ Fe + Cement + FFA	180	1.61	1.66	1.69	1.72

M → Marl
E → Electroplating waste
Fe → Steel waste

Figures 5.1 and 5.2, plotted data presented in Table 5.1 show the increase in the density of the mixtures as the binder (cement and a blend of cement and FFA) content is increased in all cases of wastes and binders. However, it is observed that the contaminated soils have significantly lesser density as compared to the uncontaminated soils. In case of the soil contaminated with steel waste, the density of the mixtures was reduced more compared to the reduction in the density of soil contaminated with electroplating waste. From Figure 5.3, it can be observed that the part replacement of the cement by FFA causes significant reduction in the density of the mixtures. This may be attributed to the fact that the FFA is lighter than cement and it requires more water for hydration as observed in the preliminary study when the FFA was blended with cement and used for preparing mixtures of marl and sand soil.

From the analysis of data pertaining to density of **marl soil mixtures containing electroplating waste**, following may be summarized:

- Increase in density due to increase in the dosage of the cement alone as binder in the range of 2.9 to 7.0% as compared to 1.8 to 3.6% increase in density due to increase in the dosage of cement and FFA used as binder.
- The decrease in density due to addition of electroplating waste was found to be in the range of 1.7 to 3.8% when cement alone was used as binder as compared to 5.7 to 9.5 % decrease in density when cement and FFA used as binder.

From the analysis of data pertaining to density of **marl soil mixtures containing steel waste**, following may be summarized:

- Increase in density due to increase in the dosage of the cement alone as binder in the range of 1.8 to 9.8% as compared to 3.1 to 6.8% increase in density due to increase in the dosage of cement and FFA used as binder.
- The decrease in density due to addition of steel waste was found to be in the range of 5.8 to 8.3% when cement alone was used as binder as compared to 8.0 to 9.0 % decrease in density when cement and FFA used as binder.

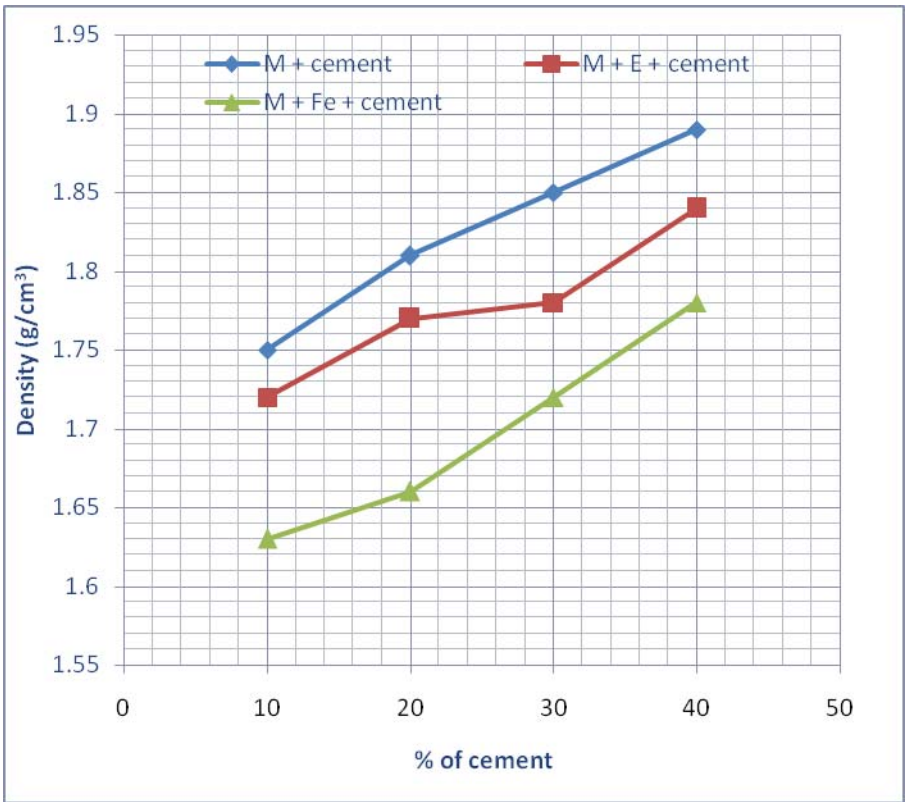

Figure 5.1 : Density of marl soil mixtures using cement alone as binder

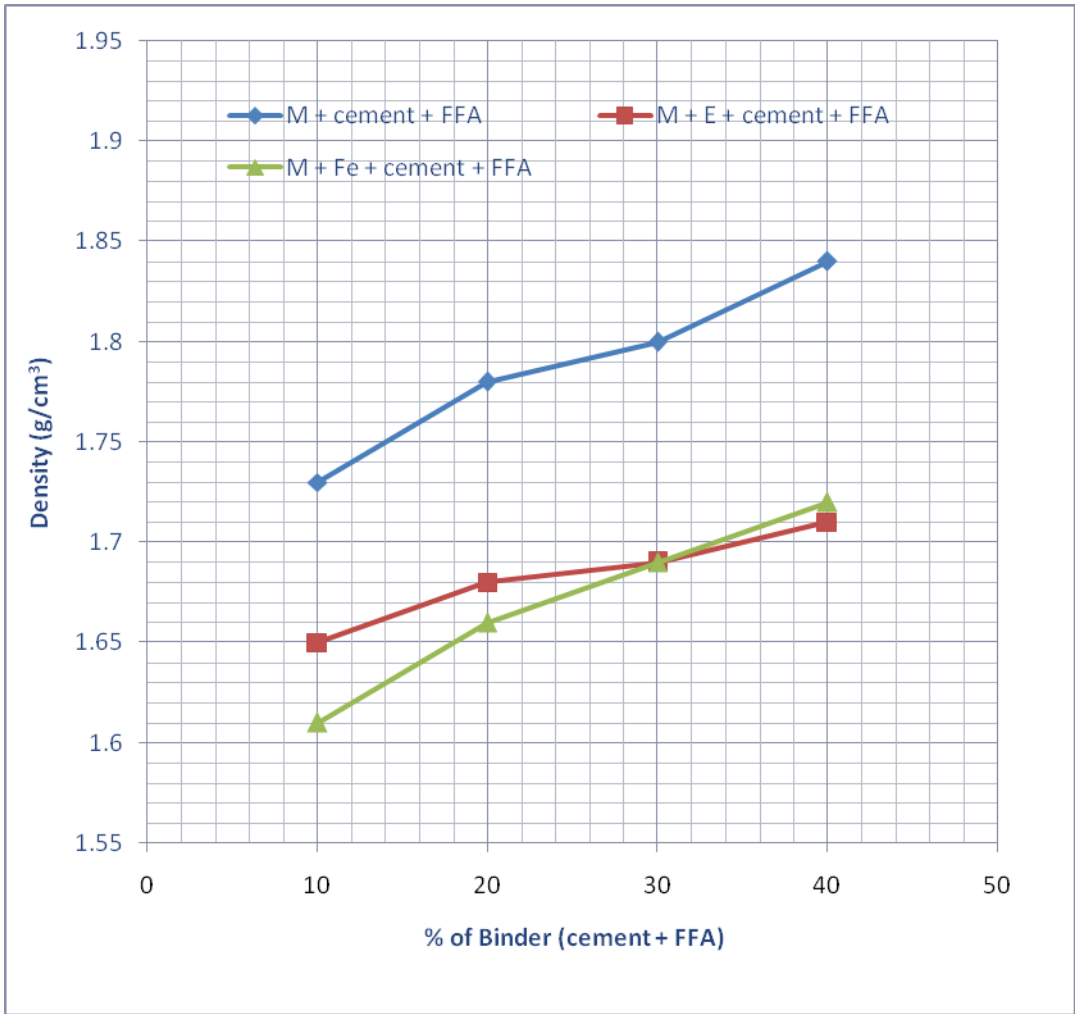

Figure 5.2 : Density of marl soil mixtures using cement and FFA as binder

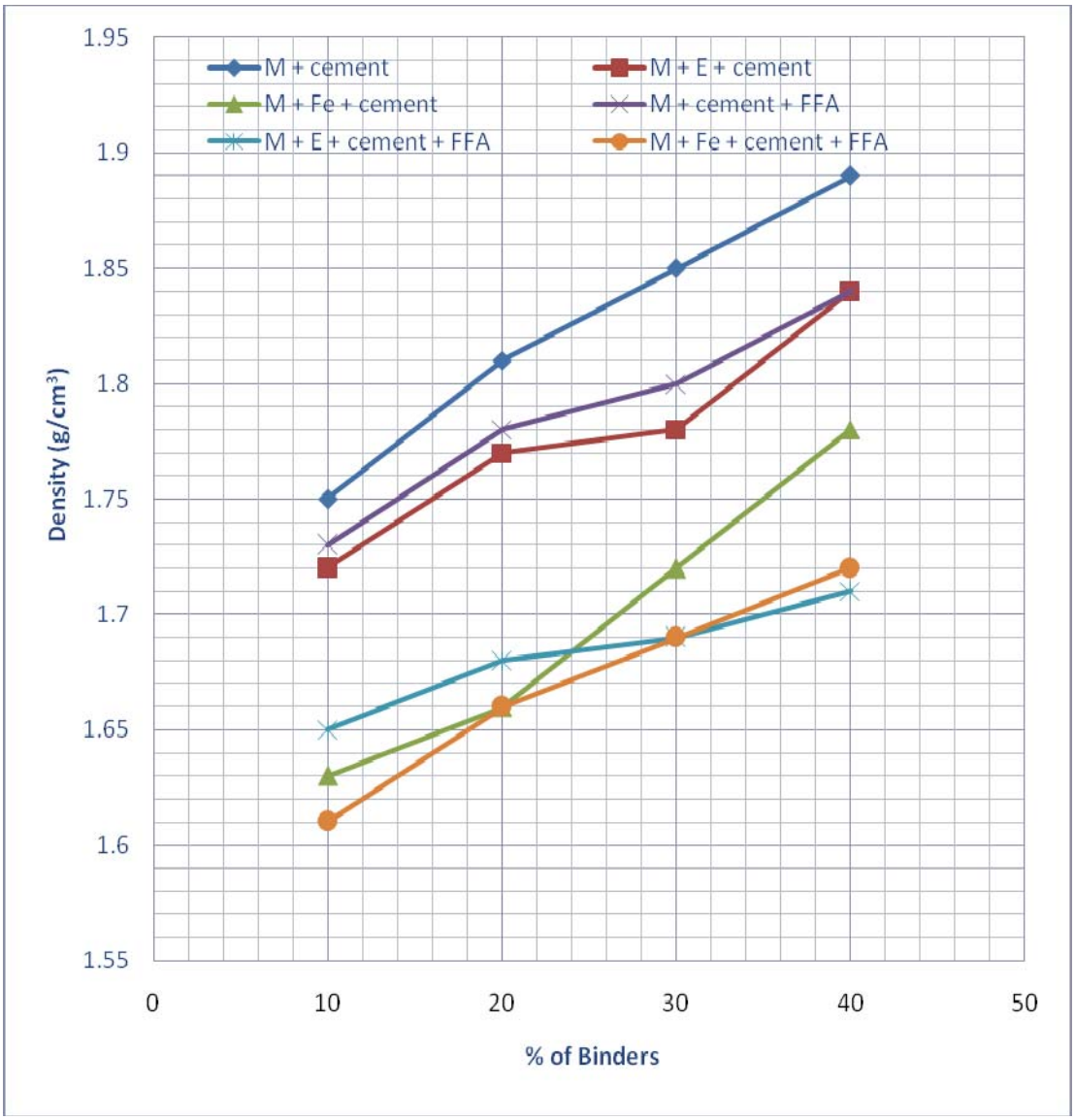

Figure 5.3 : Density of all mixtures of marl soil

5.1.2 Density of Mixtures of Sand Soil

Density obtained for the mixtures of sand soil are presented in Table 5.2. The trend as observed from the marl soil continued for the sand soil as well with the increase in the binder content, the density of the mixtures increases, the wastes reduces the density of the soil when compared to the control (Figures 5.4 and 5.5). It is very interesting to note that the decrease in the density of the mixtures of sand soil contaminated with electroplating waste is not very significant. However, in case of mixtures of sand soil contaminated with steel waste decrease in the density is very significant as can be observed from Figure 5.4. The addition of FFA as a part replacement of cement has negative effect on the density of all the mixtures except the mixture contaminated with steel waste in which case FFA has improved the density as can be seen in Figure 5.6.

As compared to the mixtures of marl soil, the mixtures of sand soil have more density in all cases of wastes and binders.

Table 5.2: Density of mixtures of sand soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		Density (g/cm^3)			
S + Cement	180	1.81	1.89	2.00	2.10
S+ E + Cement	180	1.80	1.93	2.01	2.07
S+ Fe + Cement	180	1.71	1.81	1.84	1.88
S+ Cement + FFA	180	1.76	1.80	1.86	1.92
S+ E + Cement + FFA	180	1.79	1.84	1.86	1.87
S+ Fe + Cement + FFA	180	1.75	1.82	1.85	1.87

S → Sand
E → Electroplating waste
Fe → Steel waste

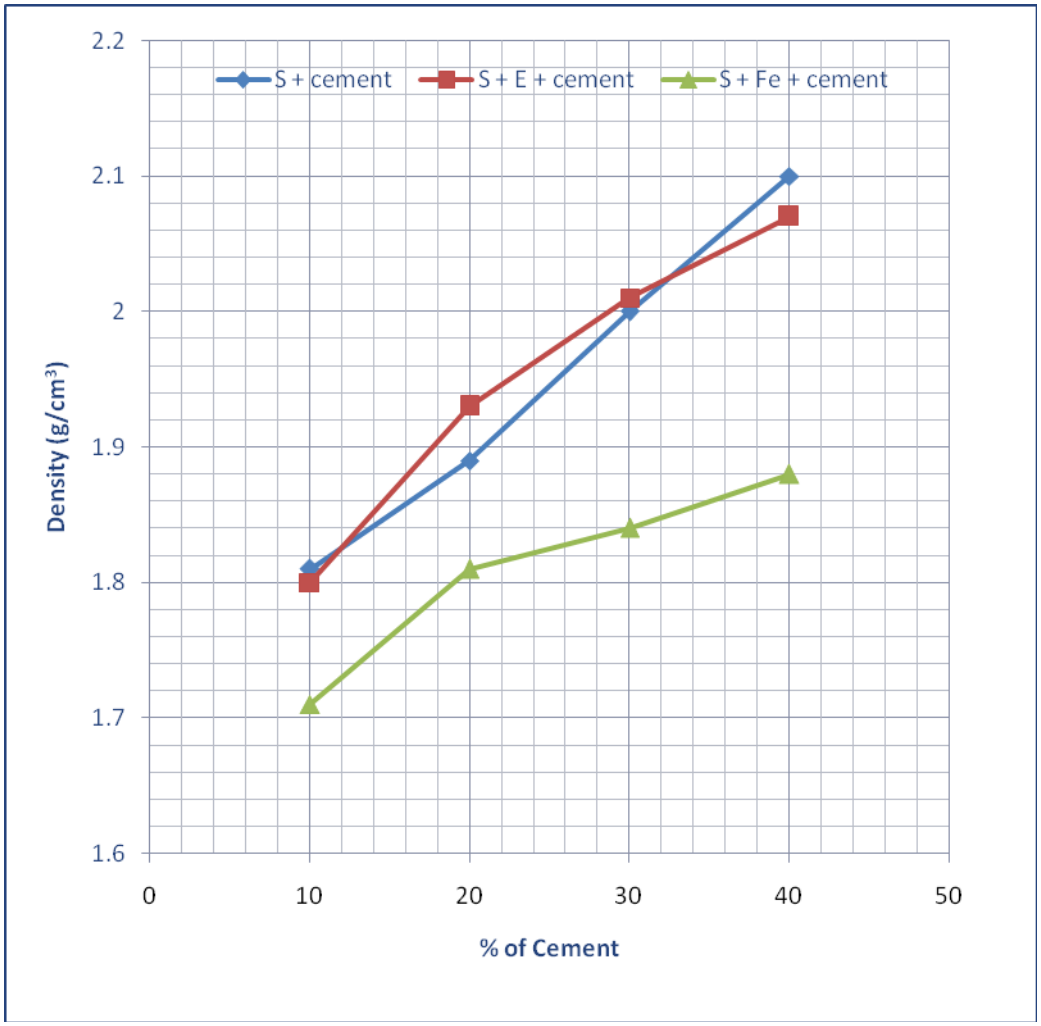

Figure 5.4 : Density of sand soil mixtures using cement alone as binder

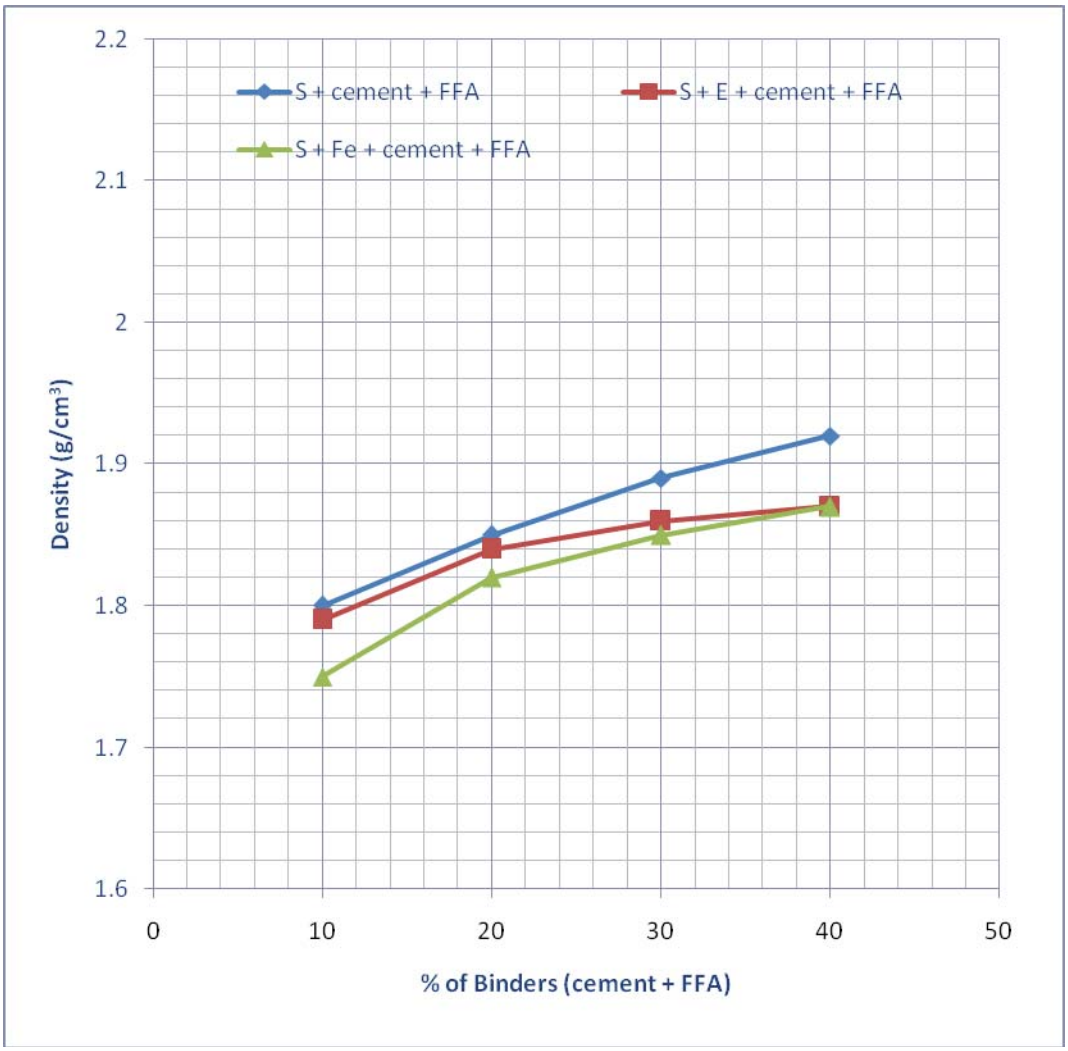

Figure 5.5 : Density of sand soil mixtures using cement and FFA as binder

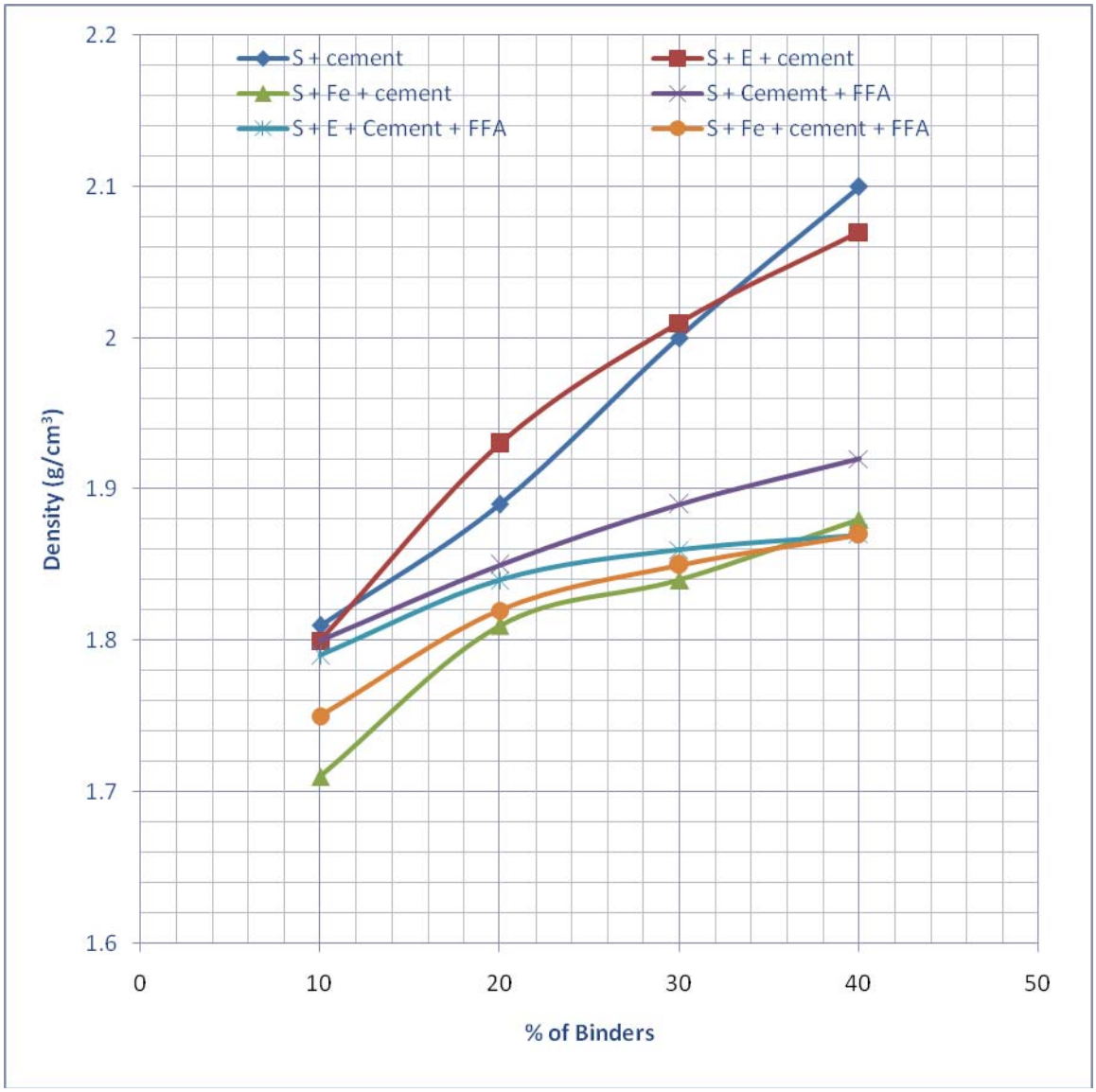

Figure 5.6 : Density of all mixtures of sand soil

From the analysis of data pertaining to density of **sand soil mixtures containing electroplating waste**, following may be summarized:

- Increase in density due to increase in the dosage of the cement alone as binder in the range of 7.2 to 15.0% as compared to 2.8 to 4.5% increase in density due to increase in the dosage of cement and FFA used as binder.
- The change in density due to addition of electroplating waste was found to be insignificant when cement alone was used as binder, however, a decrease in density in the range of 1.1 to 10.9% was found when blend of cement and FFA was used as binder.

From the analysis of data pertaining to density of **sand soil mixtures containing steel waste**, following may be summarized:

- Increase in density due to increase in the dosage of the cement alone as binder in the range of 5.8 to 10.0% as compared to 4.0 to 6.9% increase in density due to increase in the dosage of cement and FFA used as binder.
- The decrease in density due to addition of steel waste was found to be in the range of 4.2 to 10.5% when cement alone was used as binder as compared to 3.3 to 11 % decrease in density when cement and FFA used as binder.

5.2 UNCONFINED COMPRESSIVE STRENGTH (UCS)

5.2.1 UCS of Mixtures of Marl Soil

UCS test results for the mixtures of marl soil are presented in Table 5.3.

Table 5.3: UCS of mixtures of marl soil

Mixtures	Days of Curing	Dosage of Binder			
		10%	20%	30%	40%
		UCS (MPa)			
M + Cement	180	2.1	5.4	6.9	11.5
M+ E + Cement	180	2.0	4.7	6.4	11.2
M+ Fe + Cement	180	1.1	1.9	4.9	5.4
M + Cement + FFA	180	1.7	3.1	4.0	4.9
M+ E + Cement + FFA	180	1.7	2.8	4.2	4.5
M+ Fe + Cement + FFA	180	0.8	1.8	2.1	2.8

M → Marl
E → Electroplating waste
Fe → Steel waste

Figures 5.7 and 5.8, plotted using the data in Table 5.3, show the variation of UCS with binder content. There is an increase in UCS with increase in the binder content. The reduction in UCS due to electroplating waste contamination is insiginificant with cement alone as binder as well as cement blended with FFA as binder. However, steel waste has caused a significant reduction in UCS with and without FFA, as can be observed from Figures 5.7 and 5.8. It can be observed from Figure 5.9 that the addition of FFA has caused a negative effect on UCS.

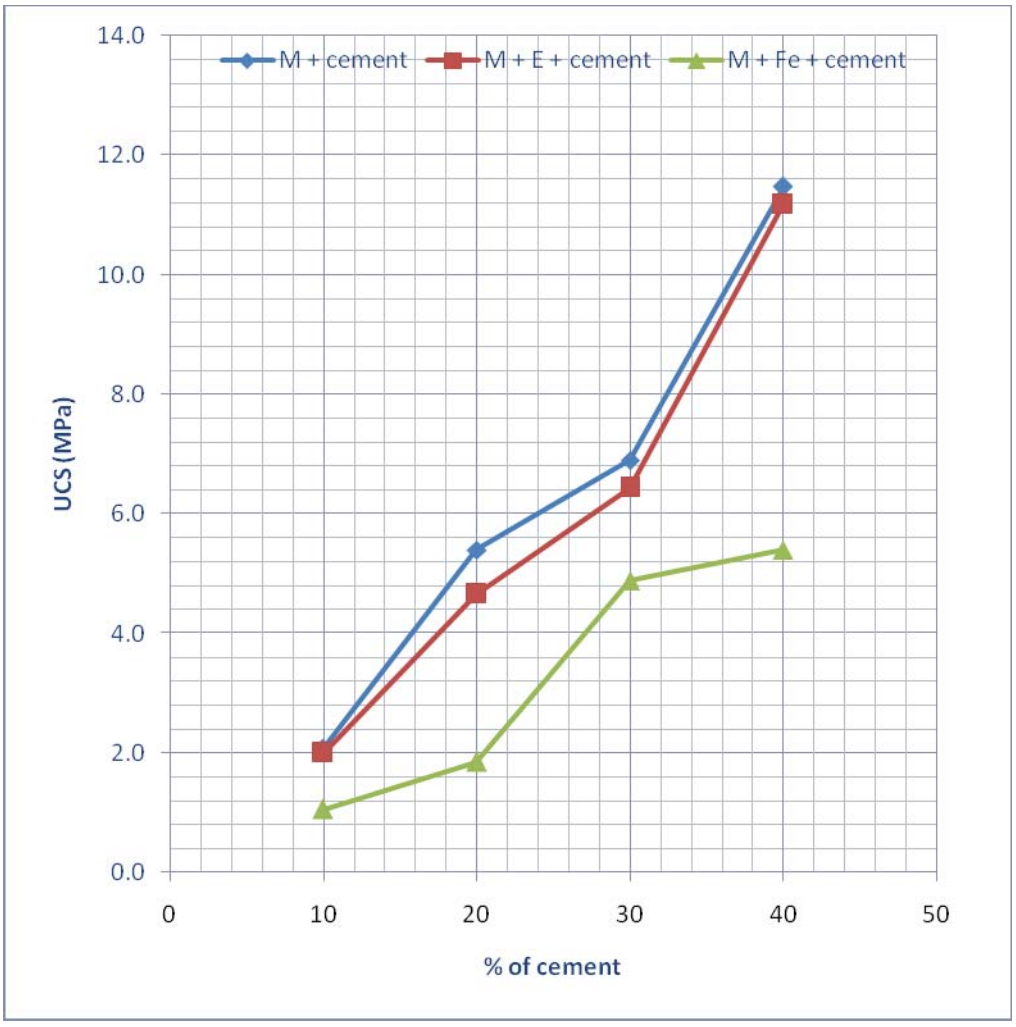

Figure 5.7 : UCS of marl soil mixtures using cement alone as binder

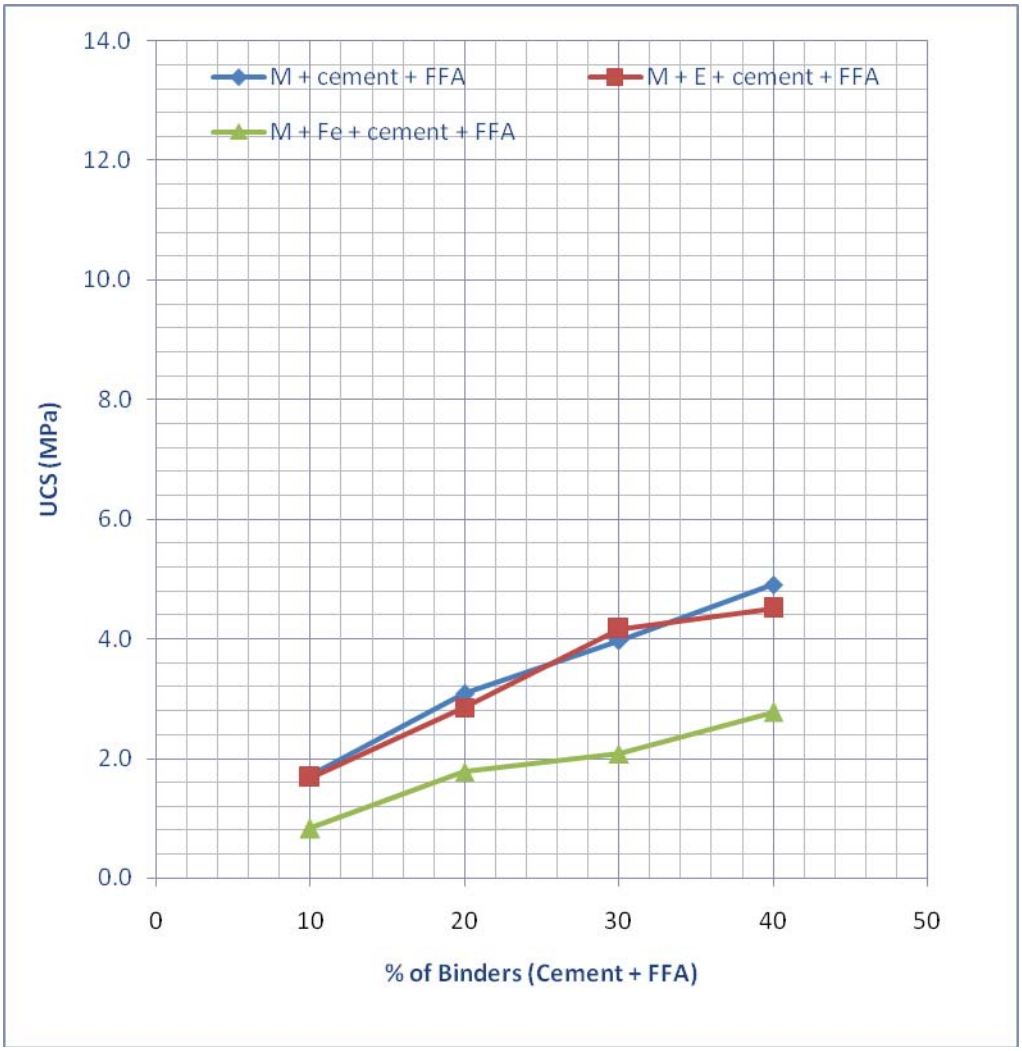

Figure 5.8 : UCS of marl soil mixtures using cement and FFA as binder

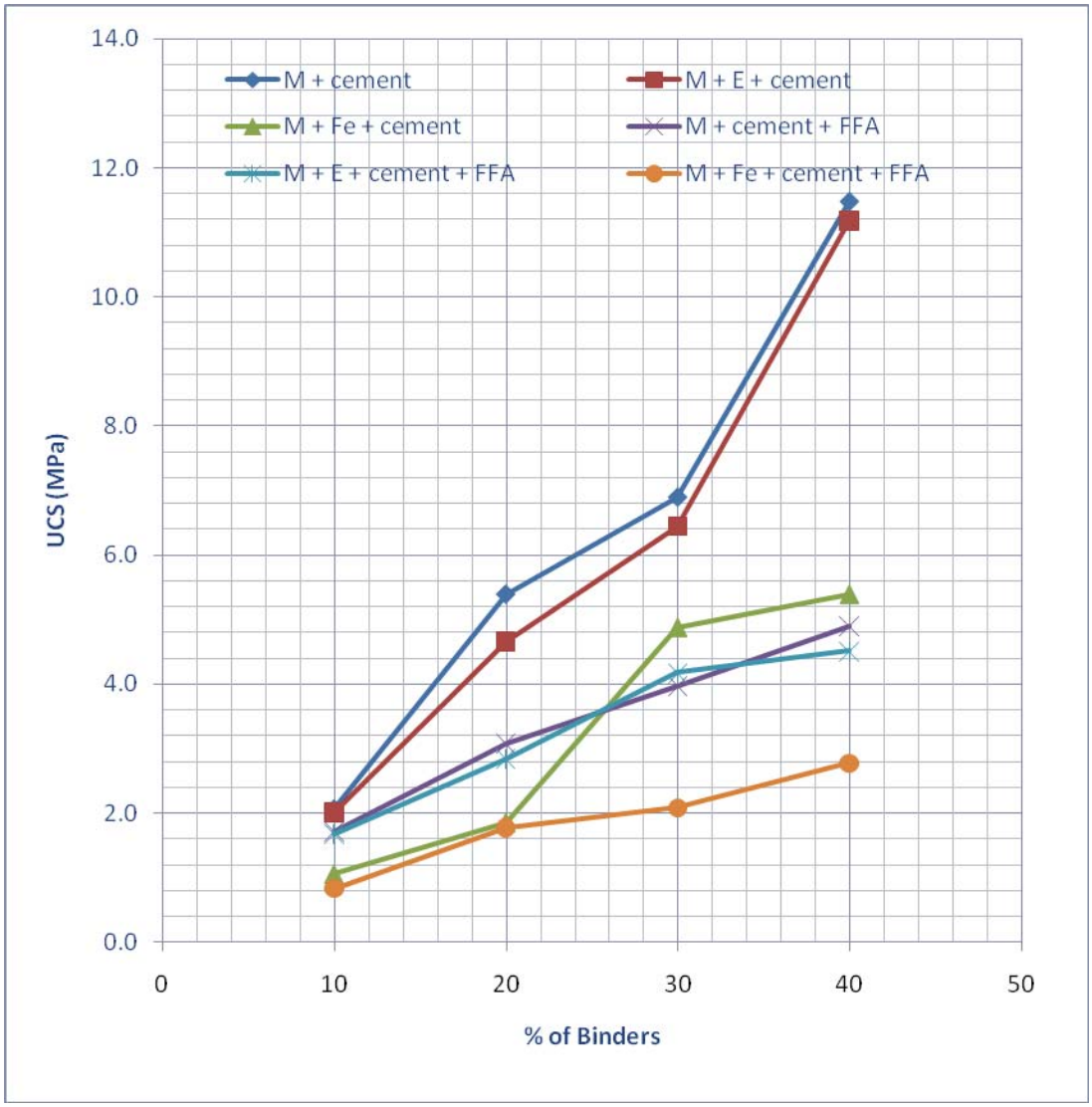

Figure 5.9 : UCS of all mixtures of marl soil

From the analysis of data pertaining to UCS of **marl soil mixtures containing electroplating waste**, following may be summarized:

- Increase in UCS due to increase in the dosage of the cement alone as binder in the range of 135 to 460% as compared to 64 to 164% increase in UCS due to increase in the dosage of cement and FFA used as binder.
- The decrease in UCS due to addition of electroplating waste was found to be in the range of 2.6 to 13.6% when cement alone was used as binder as compared to 19.0 to 60.6 % decrease in UCS when cement and FFA used as binder.

From the analysis of data pertaining to UCS of **marl soil mixtures containing steel waste**, following may be summarized:

- Increase in UCS due to increase in the dosage of the cement alone as binder in the range of 72 to 391% as compared to 125 to 250% increase in UCS due to increase in the dosage of cement and FFA used as binder.
- The decrease in UCS due to addition of steel waste was found to be in the range of 29.2 to 53.0% when cement alone was used as binder as compared to 59.9 to75.9 % decrease in density when cement and FFA used as binder.

5.2.2 UCS of Mixtures of Sand Soil

UCS values obtained for the mixtures of sand are presented in Table 5.4.

Table 5.4: UCS of mixtures of sand soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		UCS (MPa)			
S + Cement	180	3.0	6.7	11.9	15.3
S+ E + Cement	180	2.7	5.2	11.7	14.0
S+ Fe + Cement	180	0.2	0.6	2.5	3.9
S+ Cement + FFA	180	2.5	4.4	5.0	7.5
S+ E + Cement + FFA	180	2.4	4.3	4.8	7.3
S+ Fe + Cement + FFA	180	0.7	1.8	2.4	4.6

S → Sand
E → Electroplating waste
Fe → Steel waste

Figures 5.10 and 5.11, plotted using the data in Table 5.4, show the variation of UCS with binder content. There is an increase in UCS with increase in the binder content. Like the case of marl soil, the reduction in UCS due to electroplating waste contamination is insiginificant with cement alone as binder well as cement blended with FFA as binder. However, steel waste has caused a significant reduction in UCS with and without FFA, as can be observed from Figures 5.10 and 5.11 like the case of marl soil. It can be observed from Figure 5.12 that the addition of FFA has caused a negative effect on UCS in case of electroplating waste but in case of steel waste the UCS of mixtures was improved with the addition of FFA.

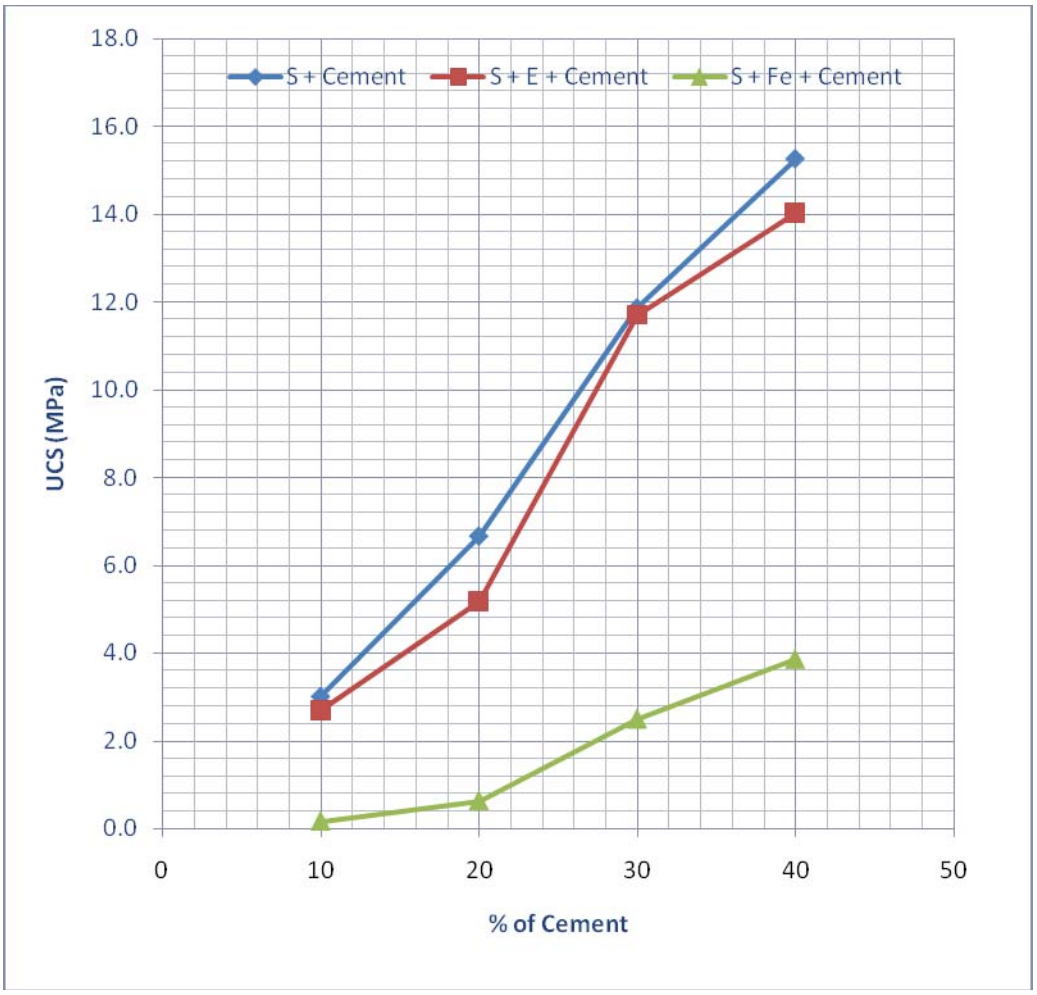

Figure 5.10 : UCS of sand soil mixtures using cement alone as binder

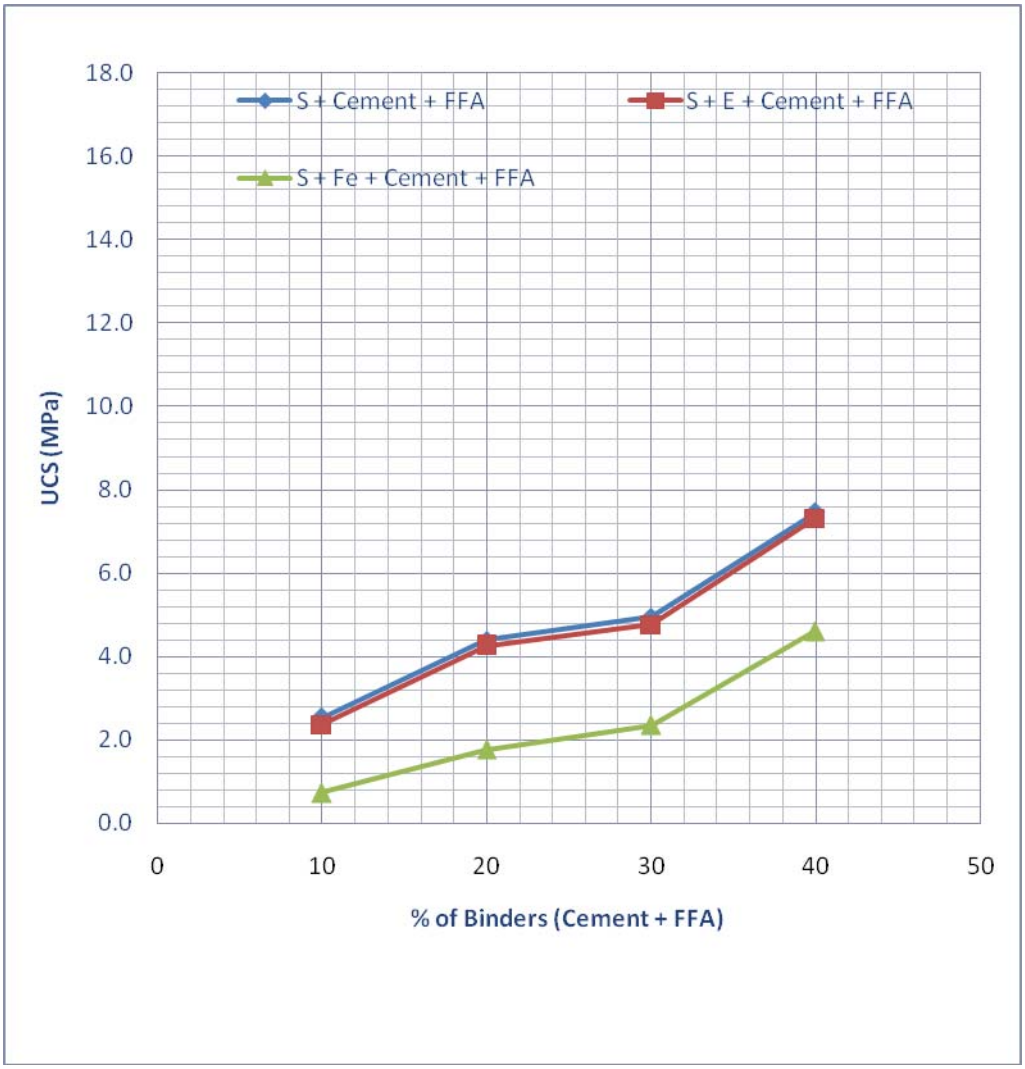

Figure 5.11 : UCS of sand soil mixtures using cement and FFA as binder

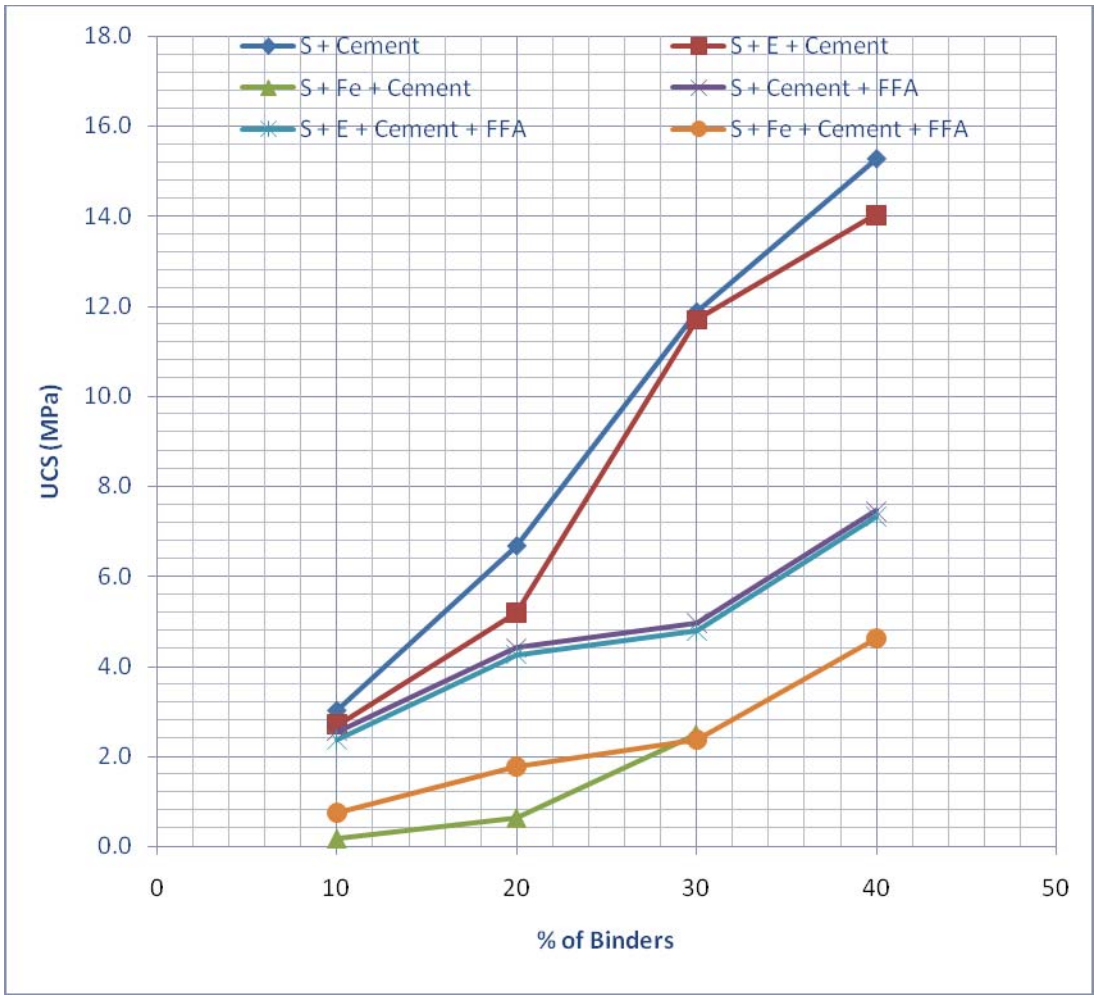

Figure 5.12 : UCS of all mixtures of sand soil

The sand combination gave the highest strength when compared to the marl soil. This is due to the texture of the sand soil in which the cement can fill in the pore spaces within the matrix of the sand and subsequent reaction to form CSH.

From the analysis of data pertaining to UCS of **sand soil mixtures containing electroplating waste**, following may be summarized:

- Increase in UCS due to increase in the dosage of the cement alone as binder in the range of 93 to 419% as compared to 79 to 204% increase in UCS due to increase in the dosage of cement and FFA used as binder.
- The decrease in UCS due to addition of electroplating waste was found to be in the range of 1.4 to 37.1% when cement alone was used as binder as compared to 21.4 to 59.8% decrease in density when cement and FFA used as binder.

From the analysis of data pertaining to UCS of **sand soil mixtures containing steel waste**, following may be summarized:

- Increase in UCS due to increase in the dosage of the cement alone as binder in the range of 200 to 1850% as compared to 157 to 557% increase in UCS due to increase in the dosage of cement and FFA used as binder.
- The decrease in UCS due to addition of steel waste was found to be in the range of 74.7 to 94.6% when cement alone was used as binder as compared to 69.8 to75.6 % decrease in UCS when cement and FFA used as binder.

5.3 POROSITY

5.3.1 Porosity of Mixtures of Marl Soil

Porosity test results for the mixtures of marl soil are presented in Table 5.5.

Table 5.5: Porosity of mixtures of marl soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		Porosity (%)			
M + Cement	180	25.74	23.11	21.69	21.00
M+ E + Cement	180	27.66	26.65	25.61	23.84
M+ Fe + Cement	180	31.20	30.99	28.90	26.74
M + Cement + FFA	180	28.13	27.00	26.39	26.19
M+ E + Cement + FFA	180	30.62	29.25	29.15	28.15
M+ Fe + Cement + FFA	180	33.98	32.31	30.55	27.68

M → Marl
E → Electroplating waste
Fe → Steel waste

As observed from Figures 5.13 and 5.14, plotted using data presented in Table 5.5, the porosity decreases as the binder content increases. However, the contaminated soils had more porosity values as compared to that of uncontaminated soils. The increase in porosity in case of steel waste is more than in case of electroplating waste. The addition of FFA has caused more porosity of the mixtures as compared to the cement alone as binder, as can be seen in Figure 5.15.

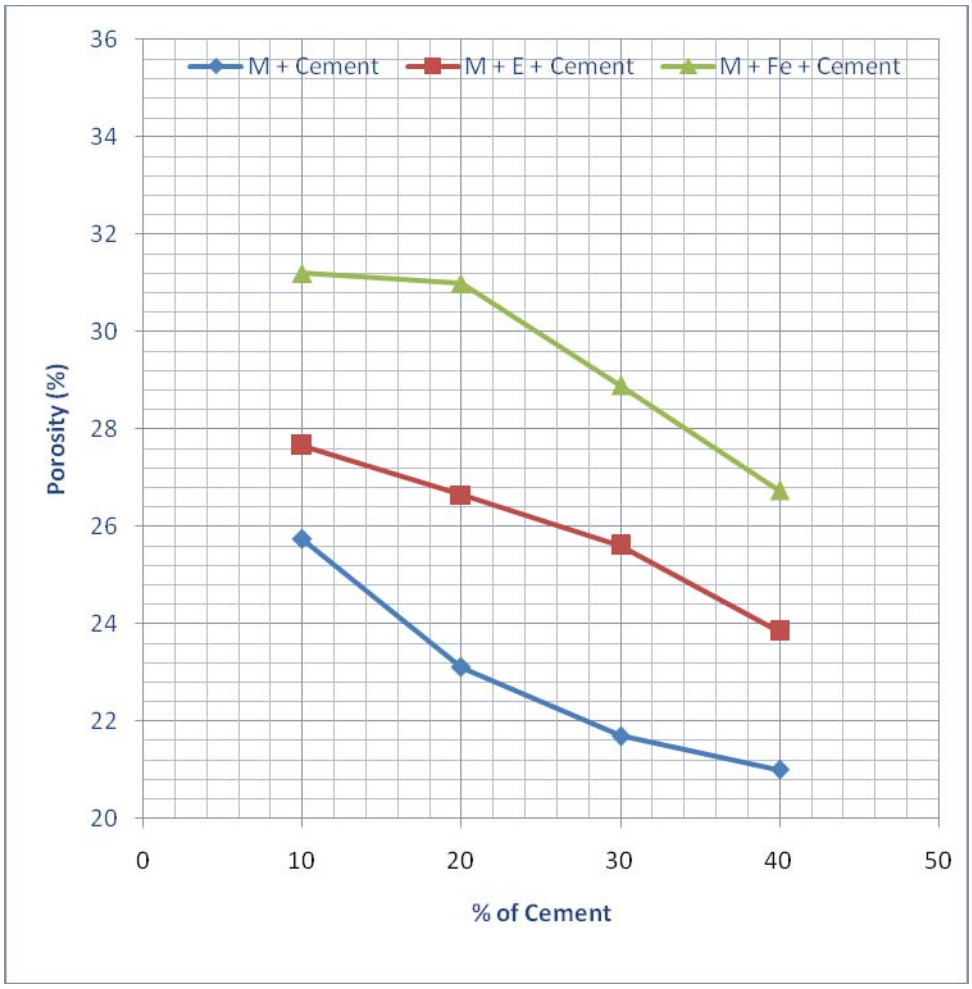

Figure 5.13 : Porosity of marl soil mixtures using cement alone as binder

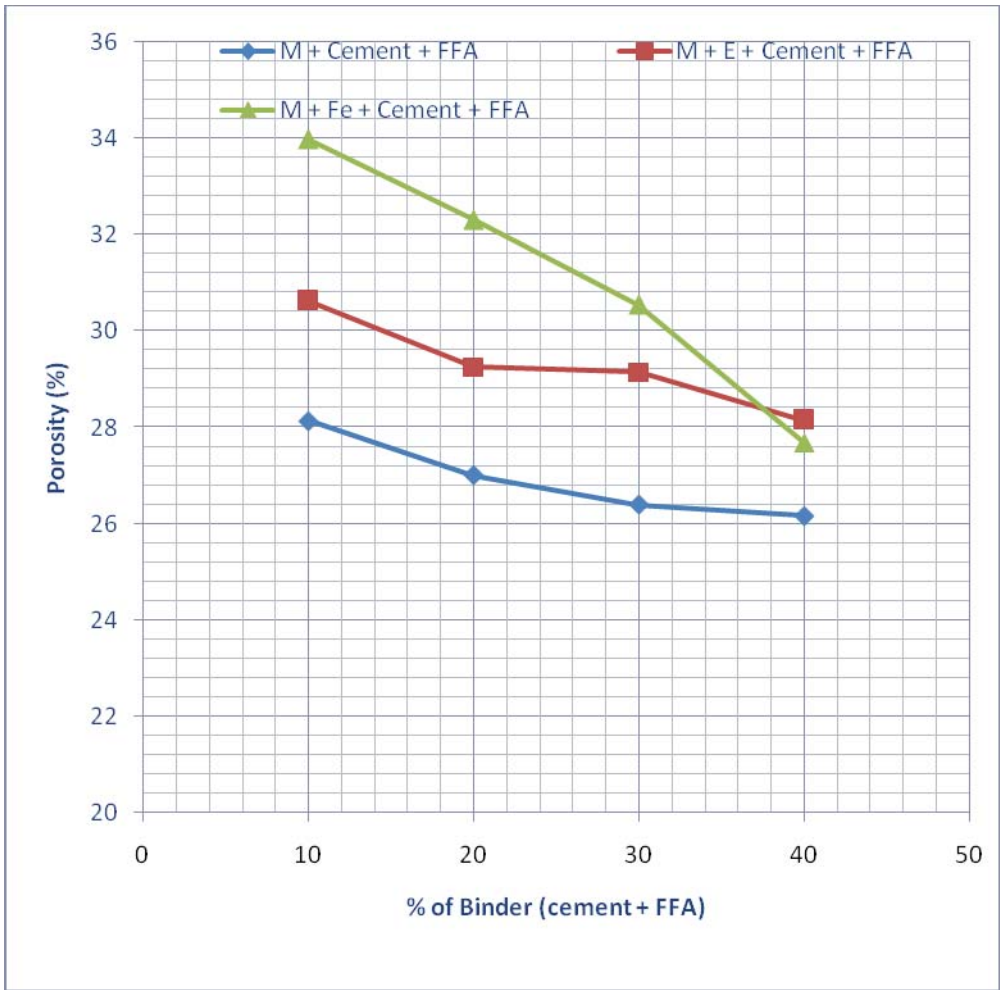

Figure 5.14 : Porosity of marl soil mixtures using cement and FFA as binder

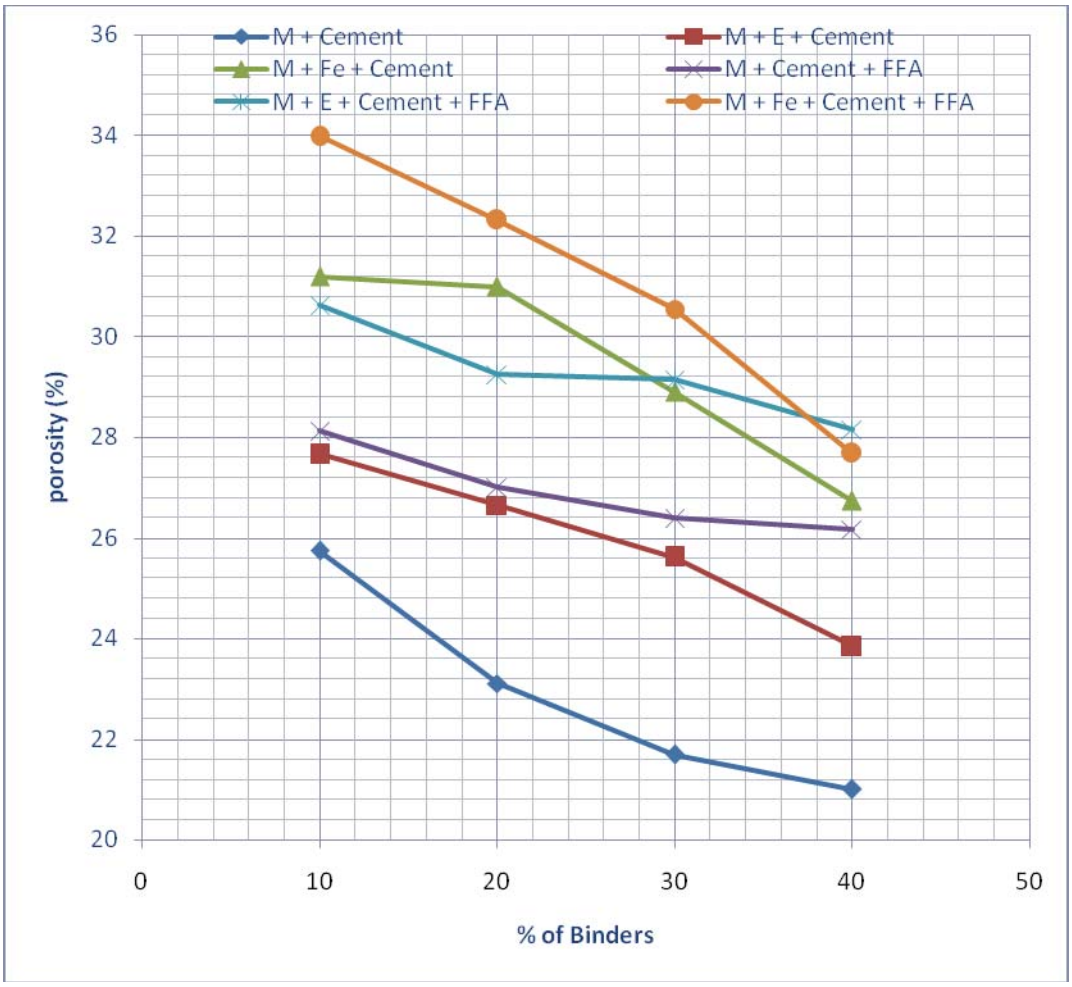

Figure 5.15 : Porosity of all mixtures of marl soil

From the analysis of data pertaining to porosity of **marl soil mixtures containing electroplating waste**, following may be summarized:

- Decrease in porosity due to increase in the dosage of the cement alone as binder in the range of 3.6 to 13.8% as compared to 4.5 to 8.1% decrease in porosity due to increase in the dosage of cement and FFA used as binder.
- The increase in porosity due to addition of electroplating waste was found to be in the range of 7.5 to 18.1% when cement alone was used as binder as compared to 19.0 to 34.3 % increase in porosity when cement and FFA used as binder.

From the analysis of data pertaining to porosity of **marl soil mixtures containing steel waste**, following may be summarized:

- Decrease in porosity due to increase in the dosage of the cement alone as binder in the range of 0.7 to 14.3% as compared to 4.9 to 18.5% decrease in porosity due to increase in the dosage of cement and FFA used as binder.
- The increase in porosity due to addition of steel waste was found to be in the range of 21.2 to 34.1% when cement alone was used as binder as compared to 31.8 to 40.8 % increase in porosity when cement and FFA used as binder.

5.3.2 Porosity of Mixtures of Sand Soil

Porosity results for the mixtures of sand soil are presented in Table 5.6.

Table 5.6: Porosity of mixtures of sand soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		Porosity (%)			
S + Cement	180	21.46	19.64	16.10	12.54
S+ E + Cement	180	21.95	17.50	13.42	13.08
S+ Fe + Cement	180	26.31	24.20	23.15	22.24
S+ Cement + FFA	180	26.57	24.55	23.51	22.06
S+ E + Cement + FFA	180	23.34	22.80	21.70	20.81
S+ Fe + Cement + FFA	180	26.05	24.11	23.59	22.47

S → Sand
E → Electroplating waste
Fe → Steel waste

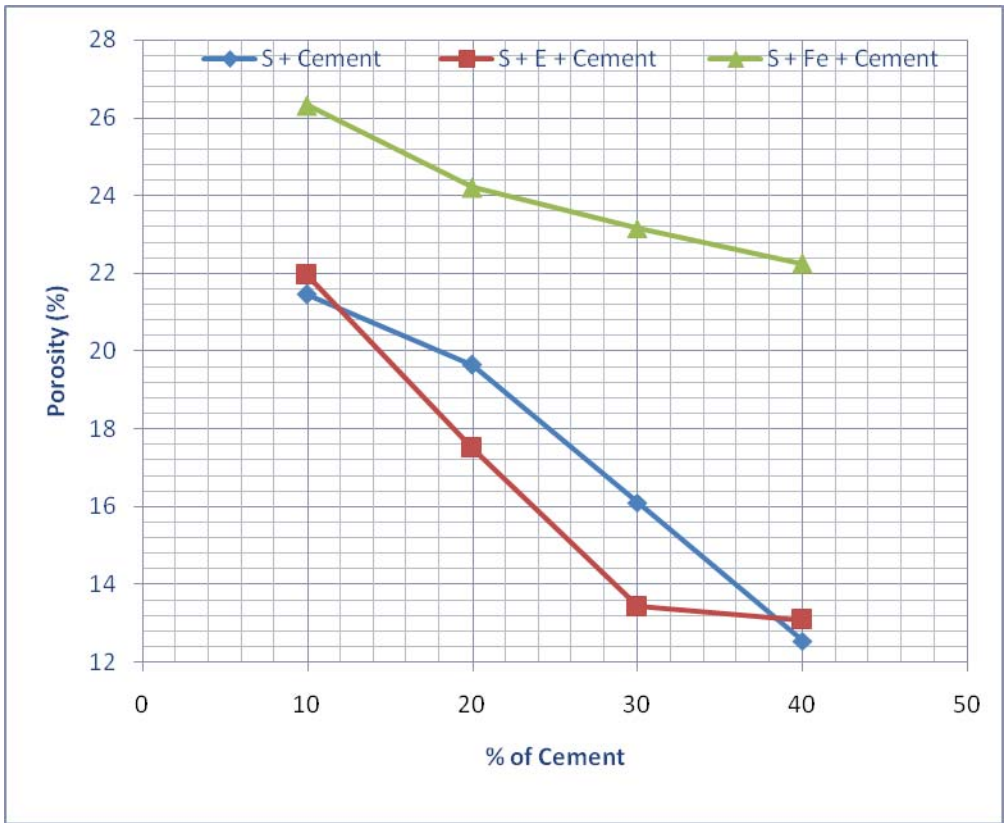

Figure 5.16 : Porosity of sand soil mixtures using cement alone as binder

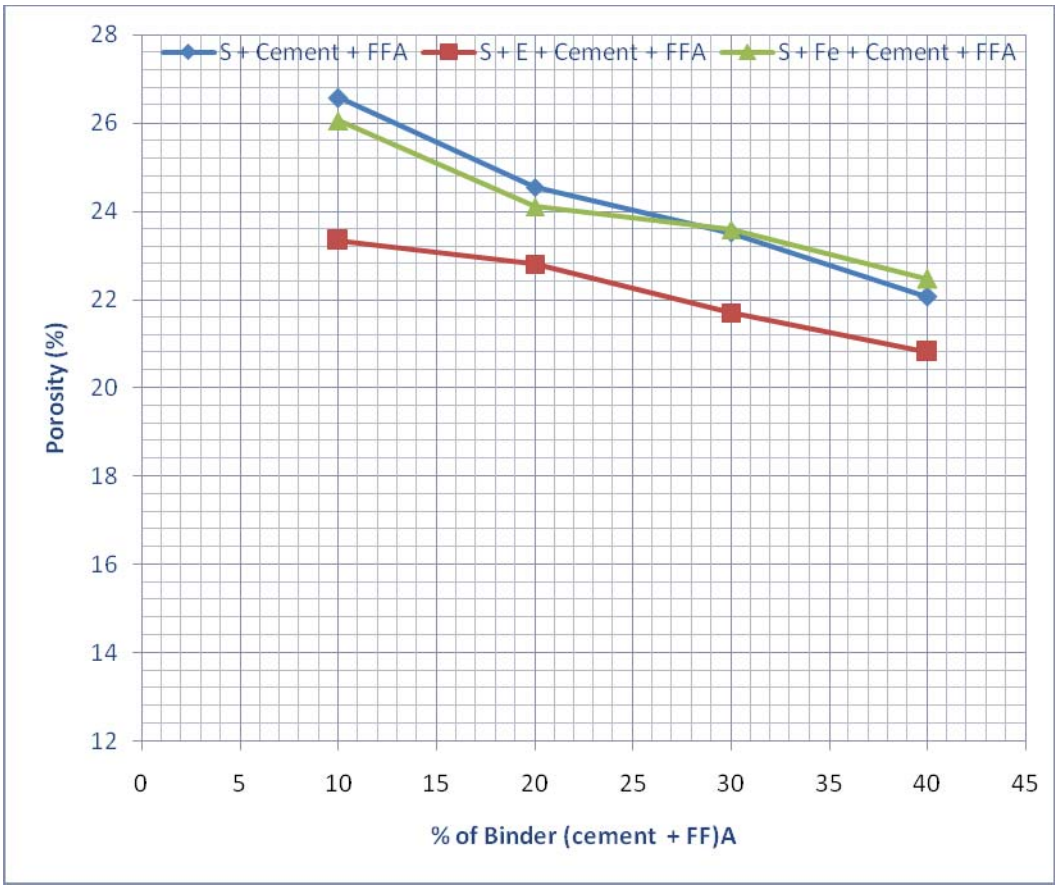

Figure 5.17: Porosity of sand soil mixtures using cement and FFA as binder

The increase in the binder caused reduction in the porosity of the mixtures as observed from the Figures 5.16 and 5.17, plotted using the data presented in Table 5.6. From Figure 5.16 it is observed that there is very significant increase in porosity due to contamination of sand soil with the steel waste whereas the porosity of sand soil contaminated with electroplating waste is not significantly affected. The addition of FFA as part replacement of cement has caused an increase in the porosity as can be seen from Figures 5.17 and 5.18. However, interestingly, with addition of FFA the effect of contamination of sand soil on porosity is almost nullified in case of steel waste and turned into positive side (i.e., porosity reduced) in case of electroplating waste.

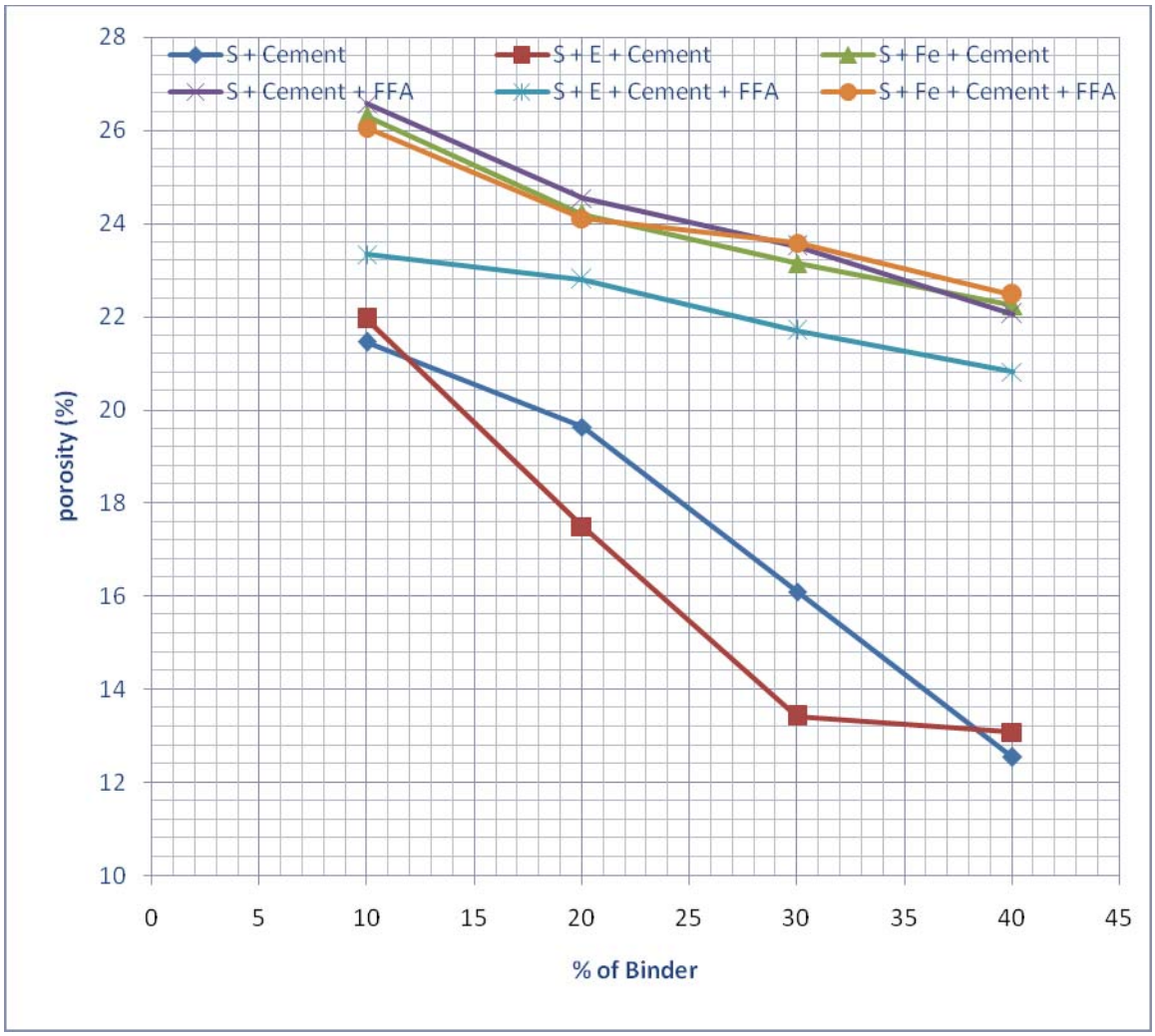

Figure 5.18: Porosity of all mixtures of sand soil

From the data on porosity of the mixtures of marl and sand soil, as presented in Tables 5.5 and 5.6, it is to be noted that the porosity of sand soil mixtures has been found to be lower than the porosity of marl soil mixtures. Further, the reduction in porosity of marl soil with increase in binder content is found to be insignificant as compared to the decrease in the porosity of sand soil with increase in the binder content.

From the analysis of data pertaining to porosity of **sand soil mixtures containing electroplating waste**, following may be summarized:

- Decrease in porosity due to increase in the dosage of the cement alone as binder in the range of 20.3 to 40.4% as compared to 2.3 to 10.8% decrease in porosity due to increase in the dosage of cement and FFA used as binder.
- The change in porosity due to addition of electroplating waste was found to be insignificant when cement alone was used as binder, however, an increase in porosity in the range of 8.7 to 65.9% was found when blend of cement and FFA was used as binder.

From the analysis of data pertaining to porosity of **sand soil mixtures containing steel waste**, following may be summarized:

- Decrease in porosity due to increase in the dosage of the cement alone as binder in the range of 8.0 to 15.5% as compared to 7.4 to 13.7% decrease in porosity due to increase in the dosage of cement and FFA used as binder.

- The increase in porosity due to addition of steel waste was found to be in the range of 22.6 to 77.4% when cement alone was used as binder as compared to 21.4 to79.2 % increase in porosity when cement and FFA used as binder.

5.4 PERMEABILITY

5.4.1 Permeability of Mixtures of Marl Soil

Permeability test results for the mixtures of marl soil are presented in Table 5.7.

Table 5.7: Permeability of mixtures of marl soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		Permeability$\times 10^{-7}$ (m/s)			
M + Cement	180	3.10	1.73	0.97	0.83
M+ E + Cement	180	7.24	4.06	3.70	3.25
M+ Fe + Cement	180	14.5	8.23	7.85	6.25
M + Cement + FFA	180	8.27	4.02	2.28	1.89
M+ E + Cement + FFA	180	12	6.40	3.57	1.16
M+ Fe + Cement + FFA	180	11.6	9.56	7.98	3.06

M → Marl
E → Electroplating waste
Fe → Steel waste

The increase in the binder caused reduction in the permeability of the mixtures as observed from the Figures 5.19 and 5.20, plotted using the data presented in Table 5.7. Soil contamination by both types of wastes has increased the permeability of the mixtures, as can be observed from Figures 5.19 and 5.20. Mixtures of marl soil including control mixtures using cement alone as binder has relatively lesser permeability than the marl soil mixtures using blend of cement and FFA as binder (Figure 5.21).

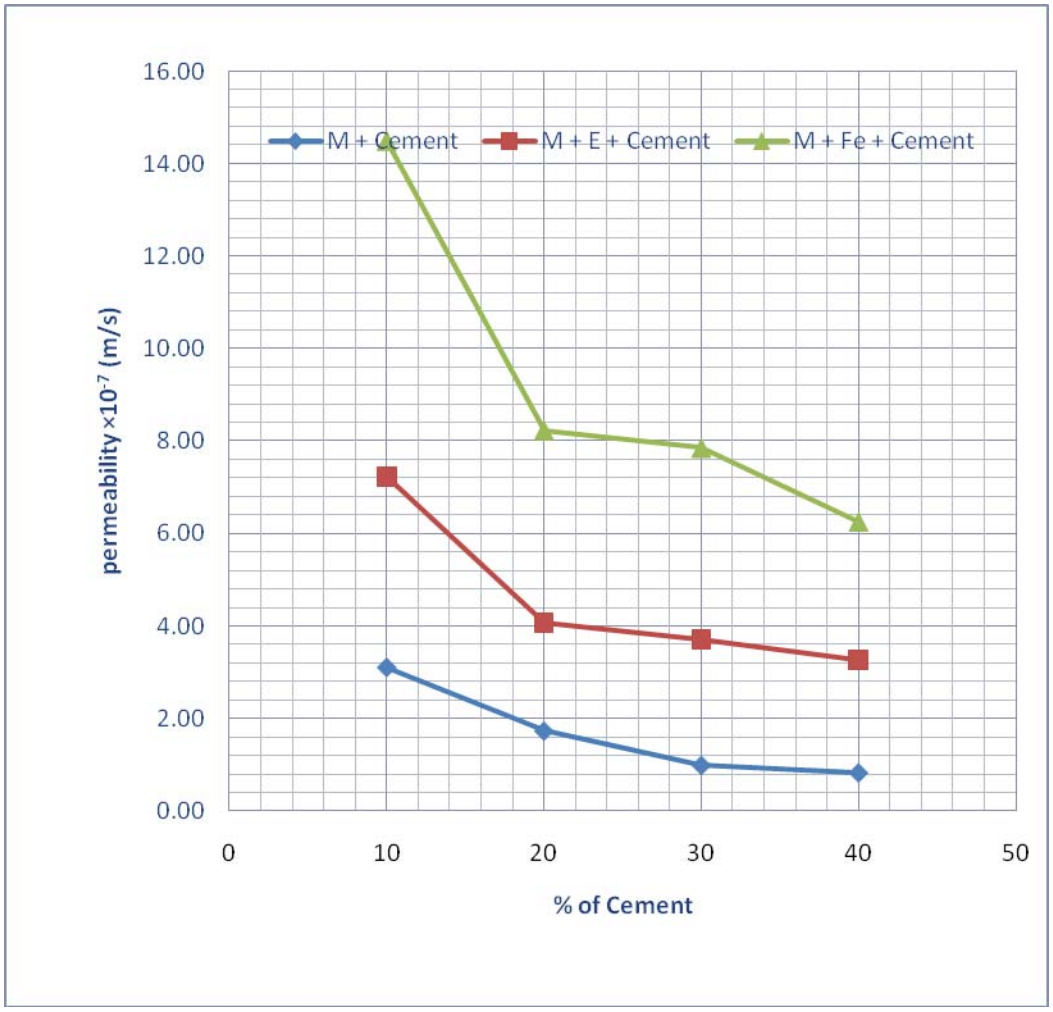

Figure 5.19: Permeability of marl soil mixtures using cement alone as binder

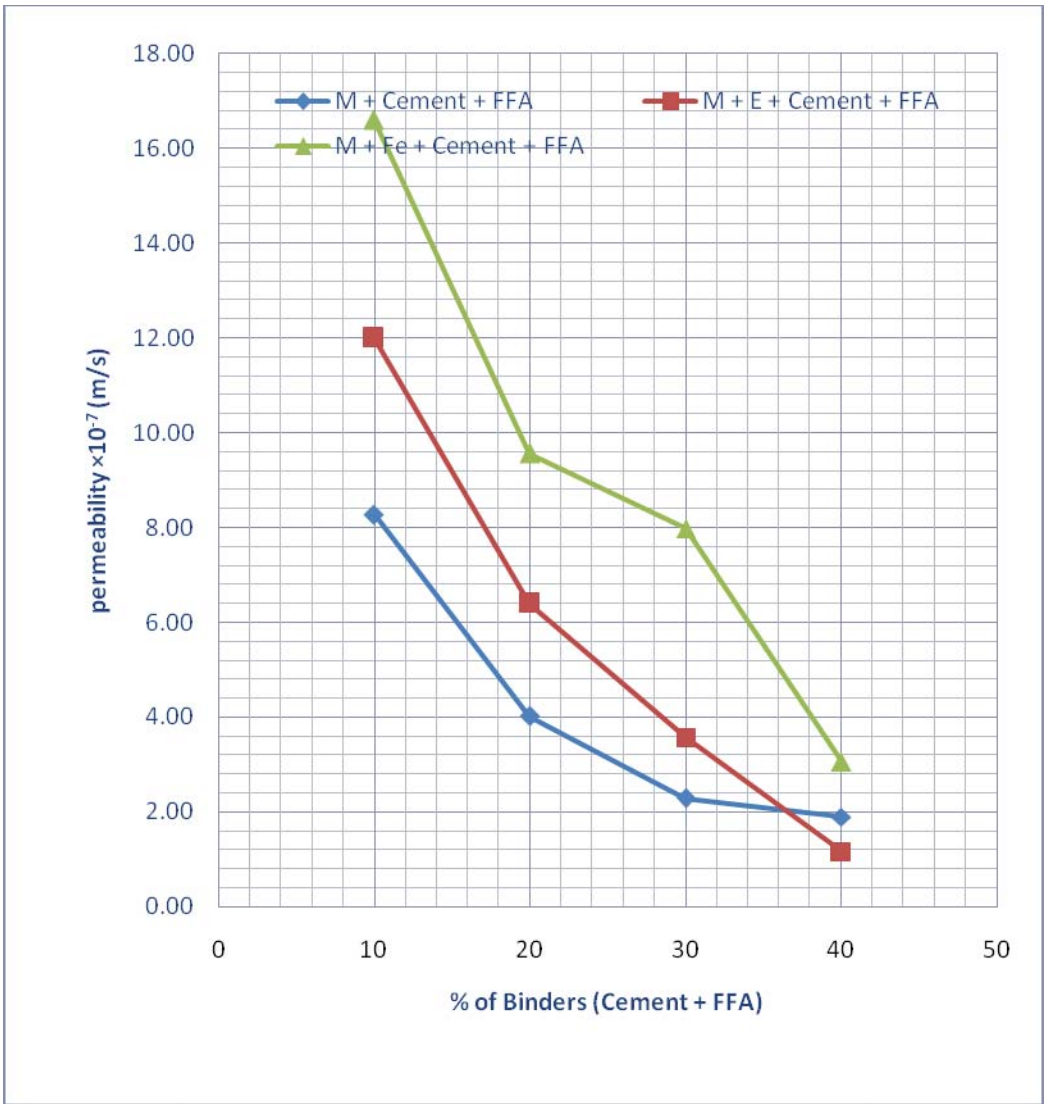

Figure 5.20: Permeability of marl soil mixtures using cement and FFA as binder

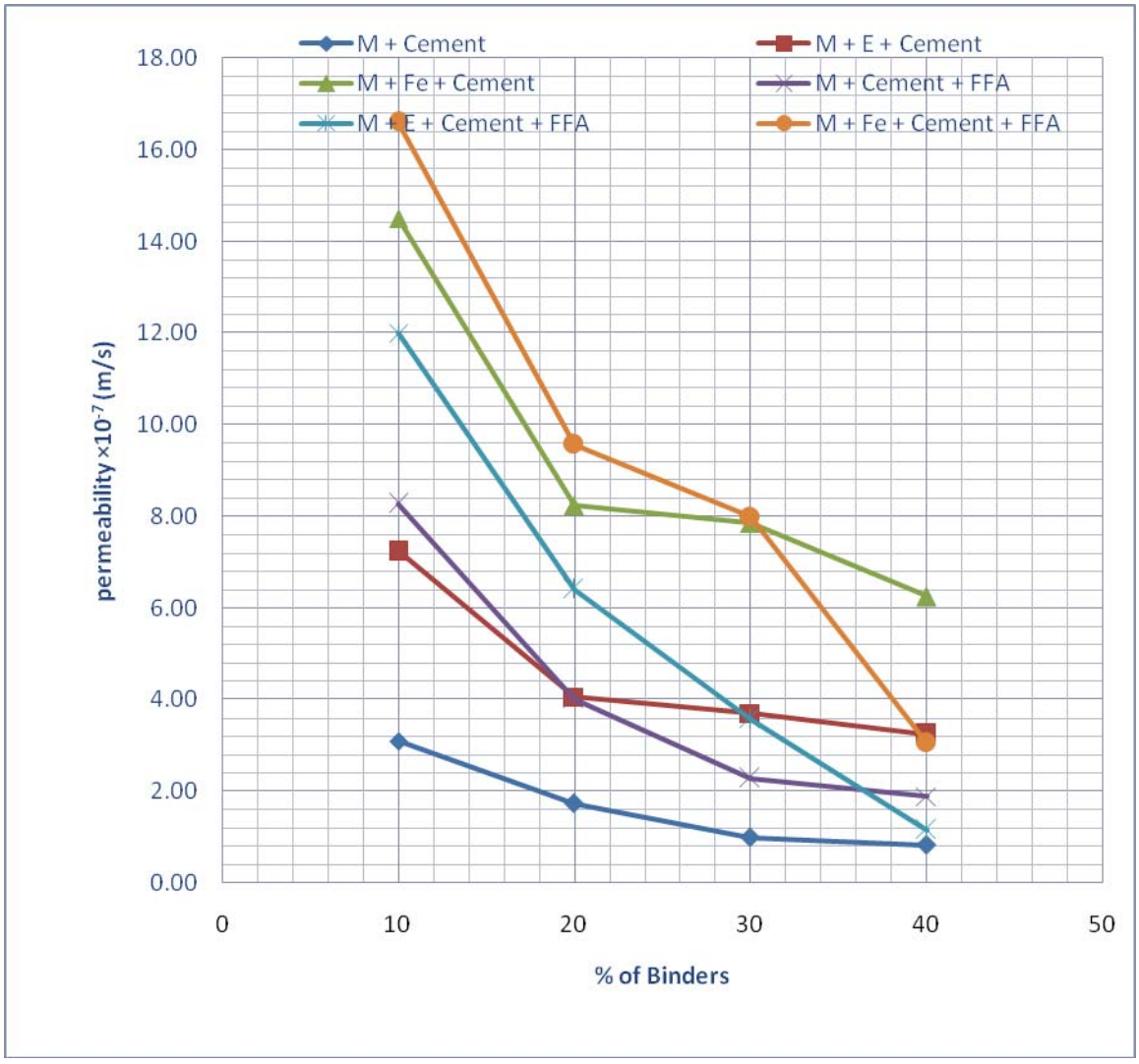

Figure 5.21: Permeability of all mixtures of marl soil

From the analysis of data pertaining to permeability of **marl soil mixtures containing electroplating waste**, following may be summarized:

- Decrease in permeability due to increase in the dosage of the cement alone as binder in the range of 44 to 55% as compared to 47 to 90.3% decrease in permeability due to increase in the dosage of cement and FFA used as binder.
- The increase in permeability due to addition of electroplating waste was found to be in the range of 134 to 292% when cement alone was used as binder as compared to 40 to 287 % increase in permeability when cement and FFA used as binder.

From the analysis of data pertaining to permeability of **marl soil mixtures containing steel waste**, following may be summarized:

- Decrease in permeability due to increase in the dosage of the cement alone as binder in the range of 43.0 to 57.0% as compared to 18.0 to 73.6% decrease in permeability due to increase in the dosage of cement and FFA used as binder.
- The increase in permeability due to addition of steel waste was found to be in the range of 367 to 709% when cement alone was used as binder as compared to 269 to 723 % increase in permeability when cement and FFA used as binder.

5.4.2 Permeability of Mixtures of Sand Soil

Permeability test results obtained for the mixtures of sand soil are presented in Table 5.8.

Table 5.8: Permeability of mixtures of sand soil

Mixtures	Days of Curing	Dosages of Binders			
		10%	20%	30%	40%
		Permeability $\times 10^{-7}$ (m/s)			
S + Cement	180	30.9	11.9	6.8	4.54
S+ E + Cement	180	28.9	12.8	7.71	1.95
S+ Fe + Cement	180	25.8	13.6	10.7	4.05
S+ Cement + FFA	180	19.4	18.4	13.2	9.94
S+ E + Cement + FFA	180	43.7	21.4	13.8	7.18
S+ Fe + Cement + FFA	180	35	18.5	11.2	7.38

S → Sand
E → Electroplating waste
Fe → Steel waste

As observed from Figures 5.22 and 5.23, plotted using data presented in Table 5.8, like the case of marl soil, permeability of sand soil also decreases as the binder content increases. The effect of waste contamination on permeability of sand soil mixtures is found to be insignificant as can be seen from Figures 5.22 and 5.23. FFA addition has caused an increase in permeability of the mixtures as compared to the case of cement alone as binder as can be seen from Figure 5.24.

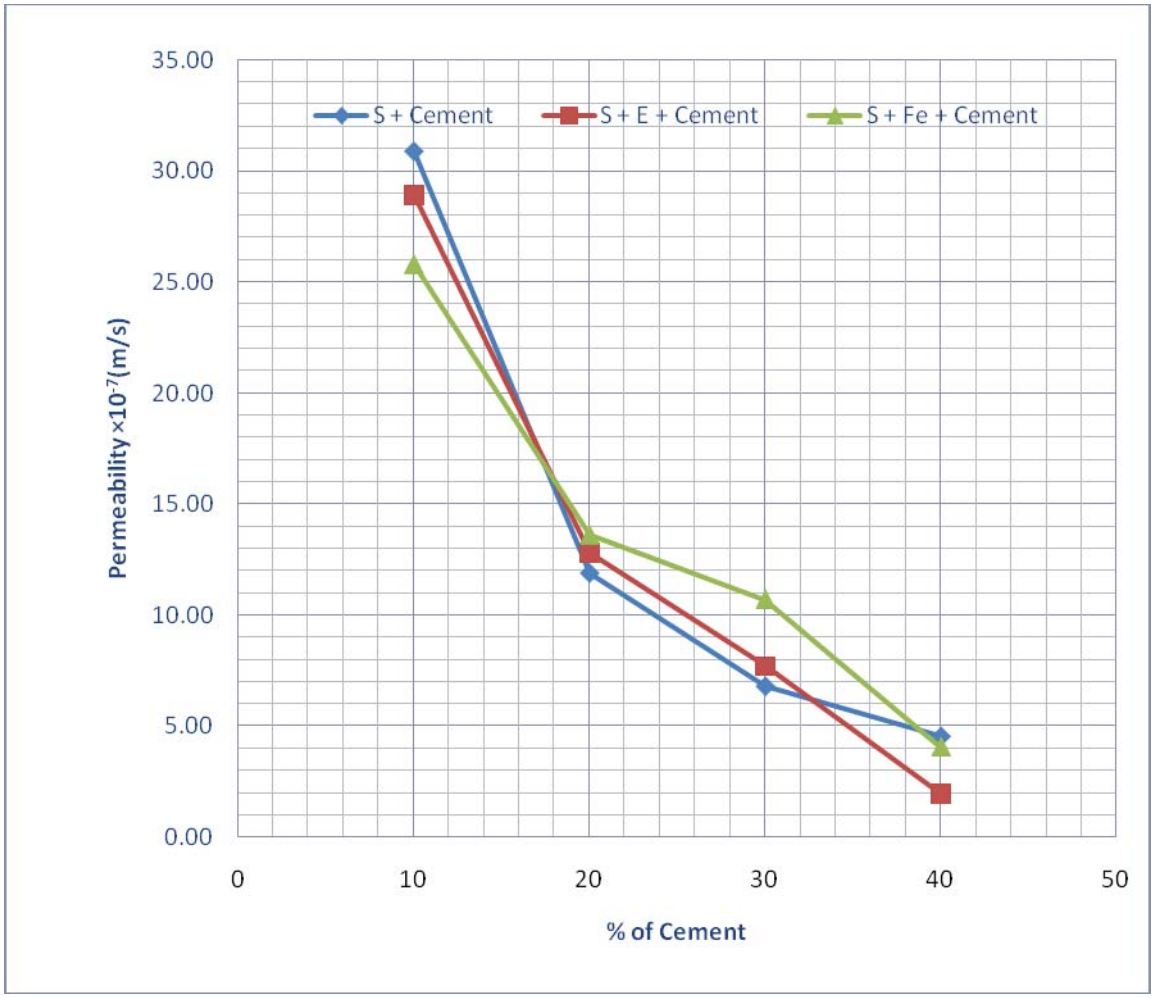

Figure 5.22: Permeability of sand soil mixtures using cement alone as binder

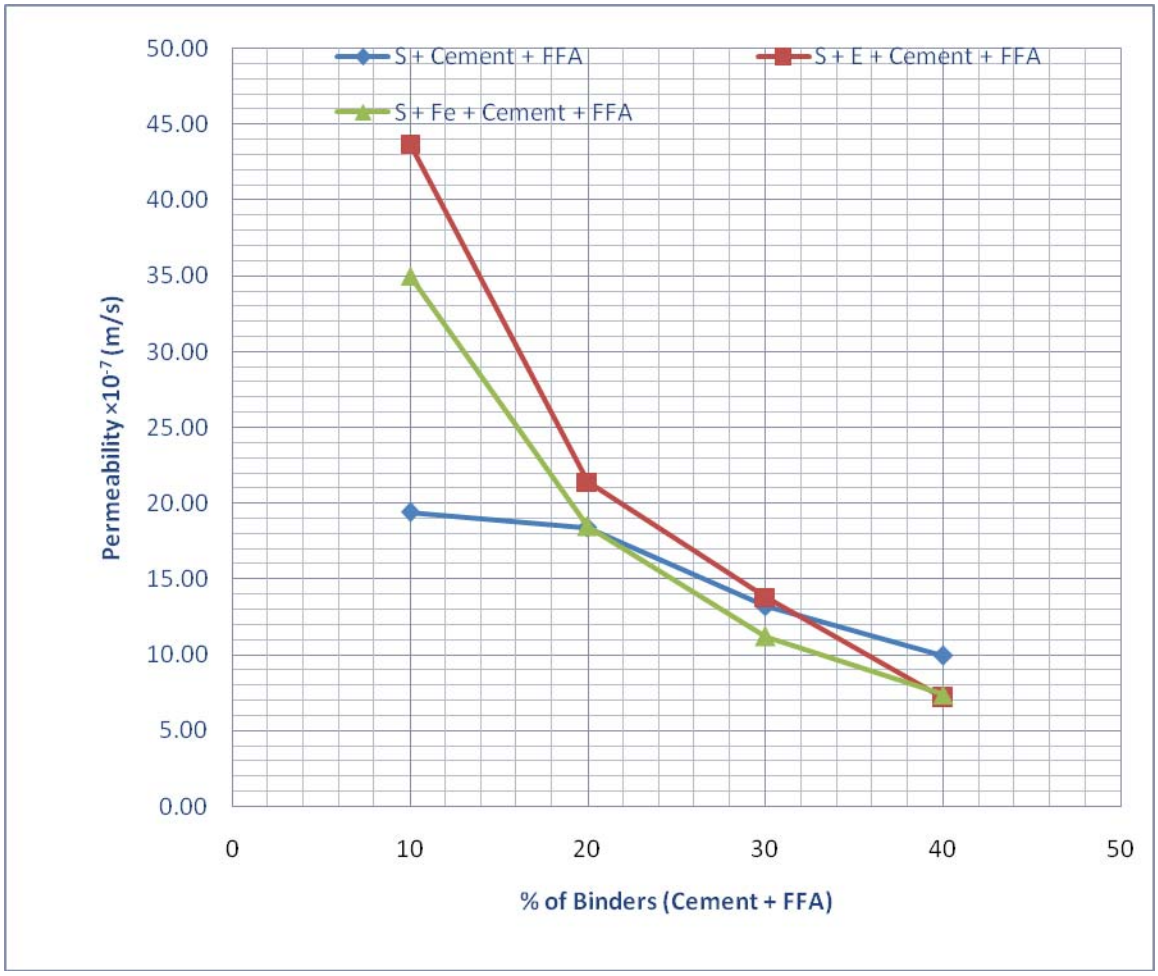

Figure 5.23: Permeability of sand soil mixtures using cement and FFA as binder

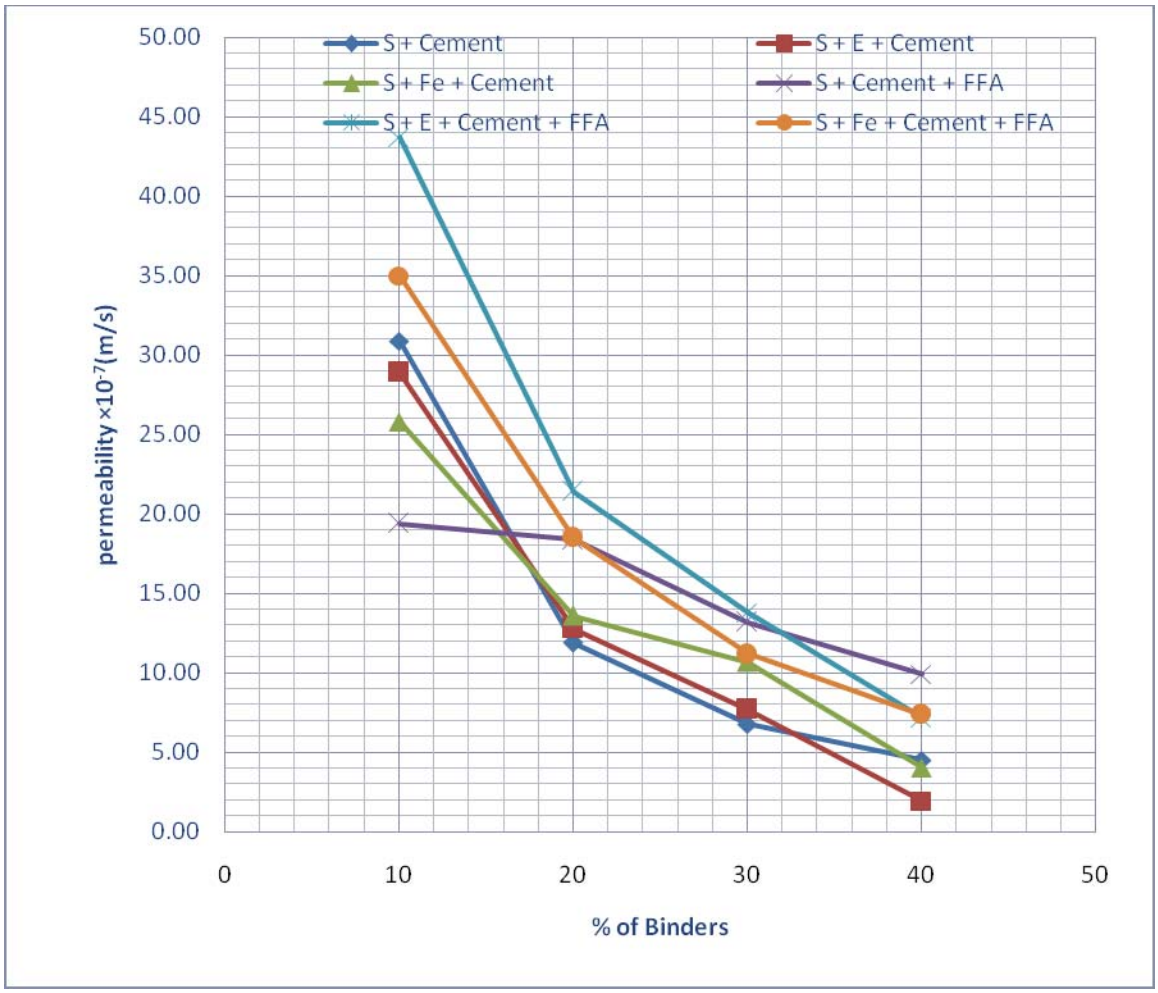

Figure 5.24: Permeability of all mixtures of sand soil

From the data on permeability of the mixtures of marl and sand soil, as presented in Tables 5.7 and 5.8, it is very interesting to note that although the porosity of sand soil has been found to be significantly lower than the porosity of marl soil, as mentioned earlier, the permeability of marl soil is found to be lower than the permeability of sand soil. The reason for this trend can be attributed to the tortuous distribution of pores in marl soil as compared to the distribution of pores in sand soil. Further, like the case of porosity, the reduction in permeability of marl soil with increase in binder content is found to be insignificant as compared to the decrease in the permeability of sand soil with increase in the binder content.

From the analysis of data pertaining to permeability of **sand soil mixtures containing electroplating waste**, following may be summarized:

- Decrease in permeability due to increase in the dosage of the cement alone as binder in the range of 55.7 to 93.3% as binder compared to 51.0 to 83.5% decrease in permeability due to increase in the dosage of cement and FFA used as binder.
- The change in permeability due to addition of electroplating waste was found to be insignificant when cement alone was used as binder, however, an increase in permeability in the range of 41.2 to 103% was found when blend of cement and FFA was used as binder.

From the analysis of data pertaining to porosity of **sand soil mixtures containing steel waste**, following may be summarized:

- Decrease in permeability due to increase in the dosage of the cement alone as binder in the range of 47.3 to 84.3% as compared to 47.1 to 78.9% decrease in permeability due to increase in the dosage of cement and FFA used as binder.
- The change in permeability due to addition of steel waste was found to be insignificant when cement alone was used as binder, however, an increase in permeability in the range of 13.3 to 64.7% was found when blend of cement and FFA was used as binder.

5.5 TOXICITY CHARACTERISTICS LEACHING PROCEDURE (TCLP)

The TCLP experiment was carried out on those mixtures which passed the acceptability criteria established by EPA for minimum strength of 0.35 MPa and maximum permeability of 10^{-7} m/s for a stabilized/solidified waste to be disposed in a landfill. Based on the TCLP results, the effectiveness of the S/S treatment of the mixtures is finally evaluated by comparing the heavy metal concentrations in the treated mixtures with their respective permissible concentrations as established by EPA .

Details of the treated mixtures that meet the criteria of UCS and permeability are presented in Tables 5.9 and 5.10, respectively.

Table 5.9 : Using the UCS criteria for marl soil and wastes

Soil type	Waste type	Stabilizers Type	Levels of stabilizers			
			10%	20%	30%	40%
Marl	Control	Cement	✓	✓	✓	✓
	Electroplating		✓	✓	✓	✓
	Steel		✓	✓	✓	✓
	Control	Cement+FFA	✓	✓	✓	✓
	Electroplating		✓	✓	✓	✓
	Steel		✓	✓	✓	✓
Sand	Control	Cement	✓	✓	✓	✓
	Electroplating		✓	✓	✓	✓
	Steel		×	✓	✓	✓
	Control	Cement+FFA	✓	✓	✓	✓
	Electroplating		✓	✓	✓	✓
	Steel		✓	✓	✓	✓

Table 5.10: Samples meeting the permeability criteria with EPA standards

Soil type	Waste type	Stabilizers Type	Levels of stabilizers			
			10%	20%	30%	40%
Marl	Control	Cement	✓	✓	✓	✓
	Electroplating		✓	✓	✓	✓
	Steel		×	×	✓	✓
	Control	Cement+FFA	✓	✓	✓	✓
	Electroplating		×	✓	✓	✓
	Steel		×	×	×	✓
Sand	Control	Cement	×	×	✓	✓
	Electroplating		×	×	✓	✓
	Steel		×	×	×	✓
	Control	Cement+FFA	×	×	×	✓
	Electroplating		×	×	×	✓
	Steel		×	×	×	✓

The mixtures selected for conducting TCLP experiment are summarized in Table 5.11.

Table 5.11: Mixtures selected for TCLP experiment.

Soil type	Waste type	Stabilizers type	Level of Stabilizers			
			10%	20%	30%	40%
Marl	Electroplating	Cement	✓	✓	✓	✓
	Steel				✓	
	Electroplating	Cement + FFA		✓	✓	✓
	Steel					✓
Sand	Electroplating	Cement			✓	✓
	Steel					✓
	Electroplating	Cement + FFA				✓
	Steel					✓

TCLP results are presented in Tables 5.12 through 5.15.

As observed from Table 5.12, all the **mixtures of marl soil containing electroplating waste** and cement alone as binder pass the TCLP criteria except the case of chromium. Same is the case when the blend of cement and FFA is used as binder except the case of the 10% dosage of the binder. Therefore, if Cr is removed by pretreatment, following are the two options for S/S treatment of the marl soil contaminated with the electroplating waste:

- S/S treatment using 10% cement as binder
- S/S treatment using 20% binder (18% cement + 2% FFA)

Table 5.12: TCLP results for marl soil (M) contaminated with electroplating waste (E)

Element	EPA Env. Standards (ppm)	TCLP Concentrations in mix samples (ppm)							
		ME1	ME2	ME3	ME4	ME5	ME6	ME7	ME8
Arsenic As	5.0	0.52	0.36	0.36	0.34	Not meeting the permeability criteria	0.36	0.18	0.18
Barium Ba	100	0.24	0.00	0.00	0.00		0.06	0.19	0.15
Cadmium Cd	1.0	0.34	0.2	0.2	0.2		0.2	0.1	0.1
Copper Cu	5.0	0.48	0.46	0.44	0.48		0.46	0.29	0.3
Chromium Cr	**5.0**	**59.72**	**49.14**	**46.5**	**32.32**		**40.88**	**30.96**	**25.54**
Lead Pb	5.0	0.073	0.063	0.054	0.043		0.3	0.14	0.14
Mercury Hg	0.2	0.34	0.12	0.12	0.12		0.3	0.09	0.12
Selenium Se	1.0	0.62	0.6	0.7	0.64		0.68	0.29	0.21
Silver Ag	5.0	0.22	0.28	0.2	0.18		1.38	1.22	0.66
Zinc Zn	10.0	2.04	0.56	0.5	0.32		0.76	0.45	0.31
	pH	8.91	8.96	9.26	10.71		8.47	8.97	9.61

ME Marl soil contaminated with electroplating waste
1-4 cement alone as binder 10-40% (i.e., 10, 20, 30, and 40%)
5-8 cement and FFA as binder 10-40% (i.e., 10, 20, 30, and 40%)

As observed from Table 5.13, following are the two options for S/S treatment of the **marl soil contaminated with the steel waste**:

- S/S treatment using 30% cement as binder
- S/S treatment using 40% binder (36% cement + 4% FFA)

Table 5.13: TCLP results for marl soil (M) contaminated with steel waste (Fe)

Element	EPA Env. Standards (ppm)	TCLP Concentrations in mix samples (ppm)							
		MFe1	MFe2	MFe3	MFe4	MFe5	MFe6	MFe7	MFe8
Arsenic As	5.0	Not meeting the permeability criteria		0.005	No TCLP conducted because at 30% binder content all metals are within limit	Not meeting the permeability criteria			0.004
Barium Ba	100			0.234					0.199
Cadmium Cd	1.0			0.006					0.006
Copper Cu	5.0			0.056					0.01
Chromium Cr	5.0			0.254					0.232
Lead Pb	5.0			0.018					0.005
Mercury Hg	0.2			0.177					0.195
Selenium Se	1.0			0.009					0.016
Silver Ag	5.0			0.027					0.039
Zinc Zn	10.0			0.732					0.237
	pH			9.000					9.29

MFe Marl soil contaminated with steel waste
1-4 cement alone as binder 10-40% (i.e., 10, 20, 30, and 40%)
5-8 cement and FFA as binder 10-40% (i.e., 10, 20, 30, and 40%)

As observed from Table 5.14, following are the two options for S/S treatment of the **sand soil contaminated with the electroplating waste** if Cr is removed by pretreatment:

- S/S treatment using 30% cement as binder
- S/S treatment using 40% binder (36% cement + 4% FFA)

Table 5.14: TCLP results for sand soil (S) contaminated with electroplating waste (E)

Element	EPA Env. Standards (ppm)	TCLP Concentrations in mix samples (ppm)							
		SE1	SE2	SE3	SE4	SE5	SE6	SE7	SE8
Arsenic As	5.0	Not meeting the permeability criteria		0.18	0.18	Not meeting the permeability criteria			0.18
Barium Ba	100			0.29	0.29				0.24
Cadmium Cd	1.0			0.11	0.11				0.11
Copper Cu	5.0			0.27	0.23				0.25
Chromium Cr	**5.0**			**28.08**	**22.69**				**29.89**
Lead Pb	5.0			0.14	0.24				0.15
Mercury Hg	0.2			0.27	0.05				0.29
Selenium Se	1.0			0.23	0.32				0.32
Silver Ag	5.0			0.16	0.07				0.13
Zinc Zn	10.0			0.83	0.21				0.48
	pH			8.55	9.12				8.40

SE Sand soil contaminated with electroplating waste
1-4 cement alone as binder 10-40% (i.e., 10, 20, 30, and 40%)
5-8 cement and FFA as binder 10-40% (i.e., 10, 20, 30, and 40%)

As observed from Table 5.15, following are the two options for S/S treatment of the **sand soil contaminated with the steel waste**:

- S/S treatment using 40% cement as binder
- S/S treatment using 40% binder (36% cement + 4% FFA)

Table 5.15: TCLP results for sand soil (S) contaminated with steel wastes (Fe)

Element	EPA Env. Standards (ppm)	TCLP Concentrations in mix samples (ppm)							
		SFe1	SFe2	SFe3	SFe4	SFe5	SFe6	SFe7	SFe8
Arsenic As	5.0	Not meeting the UCS as well permeability criteria	Not meeting the permeability criteria		0.0075	Not meeting the permeability criteria			0.009
Barium Ba	100				0.27				0.1885
Cadmium Cd	1.0				0.0075				0.008
Copper Cu	5.0				0.007				0.011
Chromium Cr	5.0				0.2265				0.2155
Lead Pb	5.0				0.012				0.006
Mercury Hg	0.2				0.14				0.1805
Selenium Se	1.0				0.009				0.01
Silver Ag	5.0				0.023				0.0235
Zinc Zn	10.0				0.1355				0.1145
	pH				10.07				9.78

SFe Sand soil contaminated with steel waste
1-4 cement alone as binder 10-40% (i.e., 10, 20, 30, and 40%)
5-8 cement and FFA as binder 10-40% (i.e., 10, 20, 30, and 40%)

The material used in the experimental program which are the marl, the sand, the cement and the FFA were tested for the hazardous metal within them so that the results can be deducted from the values obtained from the tables above. Table 5.16 gives the result of the TCLP analysis on the materials used.

Table 5.16: Results of TCLP conducted on soils and binders used in the present study

Element	EPA Env. Standards (ppm)	TCLP concentrations in samples (ppm)			
		Marl	Sand	Cement	Fuel fly ash
Arsenic As	5.0	0.023	0.007	0.000	0.562
Barium Ba	100	0.222	0.052	1.038	0.052
Cadmium Cd	1.0	0.01	0.002	0.004	0.146
Copper Cu	5.0	0.017	0.000	0.028	2.007
Chromium Cr	5.0	0.014	0.009	0.464	4.207
Lead Pb	5.0	0.007	0.009	0.01	0.161
Mercury Hg	0.2	0.000	0.000	0.041	Not Detected
Selenium Se	1.0	0.012	0.015	0.015	0.007
Silver Ag	5.0	0.005	0.000	0.009	0.001
Zinc Zn	10.0	0.028	0.000	0.000	7.541

With minimal or no presence of the heavy metals within the materials it was concluded that the baseline material used for the experimental program contain very little of the hazardous materials within the regulated standards of the EPA.

As discussed above, Cr is found to be the sole heavy metals in the soils contaminated with electroplating waste which is found to be exceeding the EPA standards even in case of S/S treatment using maximum dosage of the binders. However, blend of FFA and cement used as binder has performed better in removing Cr than the cement alone, as can be observed from Figure 5.25.

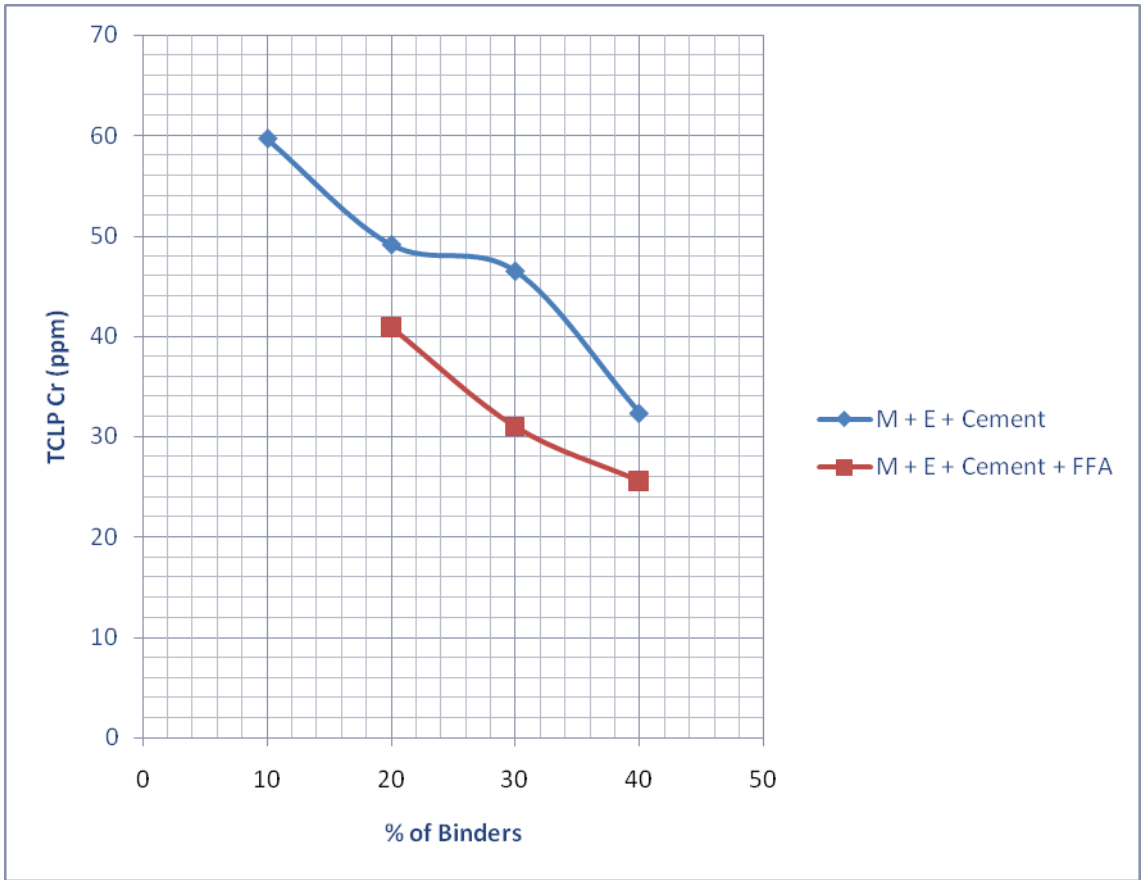

Figure 5.25: Chromium reduction on the marl soil with different binders

CHAPTER SIX

CONCLUSION AND RECOMMENDATIONS

6.1 Conclusions

From the results of the density, UCS, porosity, permeability, and TCLP tests conducted on the mixtures of various combinations of two types of soils, two types of hazardous wastes and two options of the binder for evaluating the performance of S/S treatment, following conclusions were drawn:

1) The cement alone as binder performed better than the blend of cement and FFA as binder in most of the cases. However, permeability of the treated mixtures was either slightly less or more or less same when blend of FFA and cement used as binder.
2) Blend of FFA and cement used as binder performed better than cement alone as binder in removing Cr.
3) Optimum moisture contents and FFA dosage were found for S/S treatment of contaminated marl and sand soils are as follows:
    a. 19% OMC for marl soil
    b. 12% OMC for sand soil
    c. 10% FFA for both marl and sand soils
4) The S/S treatment method was found effective in satisfying UCS and permeability criteria and also removing all heavy metals from contaminated soils within EPA Standards limits except Cr from the electroplating waste. However, except the

case of marl soil contaminated with electroplating waste, all mixtures require very high dosages of binders.

5) Dosages of binders required for simultaneously meeting UCS, permeability, and TCLP criteria were found to be as follows:
    a. 10% (cement alone) for marl soil contaminated with electroplating waste, subject to the removal of Cr using some pretreatment
    b. 30% (cement alone) for marl soil contaminated with steel waste
    c. 30% (cement alone) for sand soil contaminated with electroplating waste, subject to the removal of Cr using some pretreatment
    d. 40% (cement 36% + FFA 4%) for sand soil contaminated with steel waste

6.2 Recommendations for Future Research

The following recommendations can be made for further research in this area

1) Investigate immobilization technique for the marl and sand soil contaminated with waste by carrying out the X-Ray Diffractometer (XRD) one the stabilized/solidified mass.
2) Use of higher percentage of FFA as part substitute for cement but not jeopardizing the strength and the permeability criteria.
3) There is a need for finding ways and means for removal of Cr before S/S treatment of soil contaminated with a waste containing very high concentration of chromium (Cr)
4) This study is limited to size of specimen, there is need to look at having a length of about 2 m so that the tensile strength can be conducted on samples, to prevent breakage when placed in the landfill after a long term.

5) There is a need to look at the overall effect of higher amount of cement on the S/S material to prevent cracks being formed on the long term when the S/S material is placed in the landfill.
6) The economy of placing the hazardous contaminated soil in clay liner landfill or membrane liner landfill instead of using higher percentage of cement to stabilize/solidify.

APPENDIX A

Determination of Brine Density

Procedures

1. 50 g of the salt is dissolved in 1000 ml of de-ionized water and stirred with magnetic stirrer for 10 minutes so that all the salt dissolved completely.
2. Weigh an empty pycnometer, with volume of 10 cm^3, and record the weight
3. Fill the pycnometer with the brine solution and take the weight full of the pycnometer and record the weight.
4. Determine the weight of the solution by subtracting the weight of the empty pycnometer from the weight full of the solution.
5. Divide the weight obtained by the volume of the pycnometer to obtain the density.

Calculations

Weight of empty pycnometer = 15.423g
Weight of full pycnometer = 25.795g
Weight of solution = 10.363g

$$\text{Density } \rho_f = \frac{10.363g}{10cc} = 1.0363g/cm^3$$

Determination of Brine Viscosity using Oswald Viscometer

Procedures

1. Wash the viscometer with an organic solvent
2. The brine is then suck into the bulb of the viscometer and then allowed to flow under its own weight

3. Time it takes for the brine level to flow from upper fiducial mark to lower fiducial mark is recorded as shown in Figure A1

4. Calculate the kinematic viscosity, from here, the dynamic viscosity of the brine can be calculated

Calculation

Viscosity No y308
size 100
C AT 100^0F 0.01515
C AT 210^0F 0.01507

C AT $1^0F = \frac{0.01515\text{-}0.01507}{210\text{-}100} = 7.2727\times10^{-7}$

C AT $75^0F = 0.01515 + 7.2727\times10^{-7}\times(100-75)$

= 0.0151682 C.Stokes/sec

Time recorded was 68.78 secs

kinematic viscosity, $\nu = 0.0151682\times68.78$

= 1.04327c.Stokes

Dynamic viscosity, $\mu = \nu\times\rho$

$= 1.04327\times1.0363 = 1.08112$ c. poise

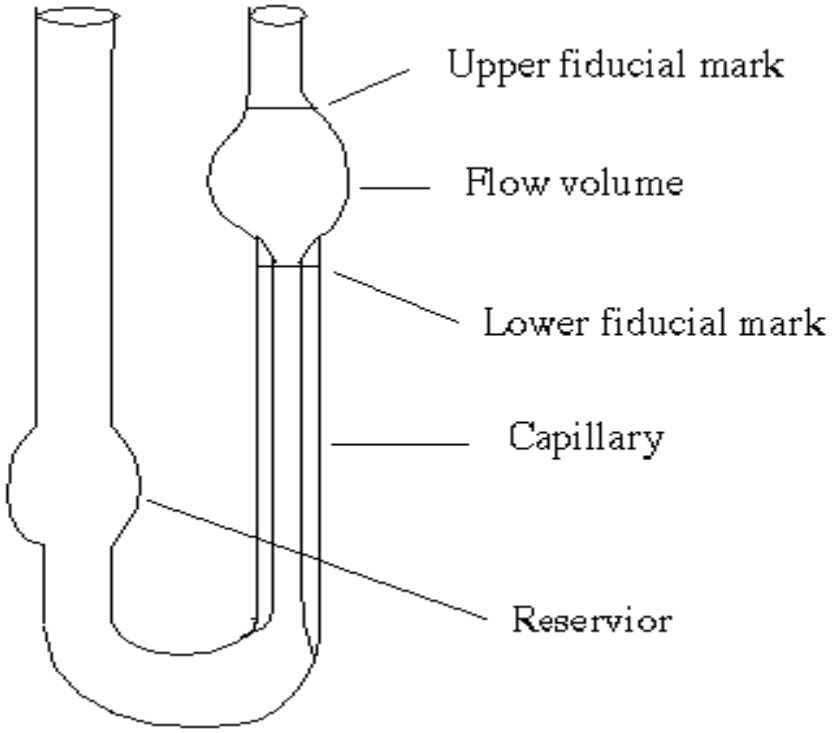

Figure A.1: Oswald viscometer

APPENDIX B

Typical example for permeability test

Sample No.: 38

Atmospheric Pressure Pa (psia): 14.7

Length, L (cm): 7.62

Brine Concentration, ppm 50000

Diameter, D (cm): 3.78

Room Temperature, Tr (^{o}C)

Cross-Sectional Area, A (sq.cm): 11.22

Brine Viscosity, μ_b (cp): 1.08112

Barometer Reading, Ha (mm. Hg): 760.7

Table B.11: Permeability Calculation

Atmospheric pressure	Displacing pressure	Differential Pressure $\frac{P_d\,(psig)}{P_a\,(psia)}$	Volume of brine collected	Time of collecting V_b	Average flow rate $\bar{Q} = \frac{V_b}{t}$	Slope, m $\frac{\Delta Q}{\Delta P}$	Liquid Permeability
P_a (apsi)	P_d (psig)	$\Delta P(atm)$	V_b (cc)	t (sec)	cc/sec	cc/sec/atm	$K(md)$
14.7	8.1	0.55	10	534.4	0.0187	0.108	79.28
14.7	10.5	0.71	10	290.68	0.0344		
14.7	12.6	0.86	10	194.56	0.0514		
14.7	15	1.02	10	144.45	0.0692		

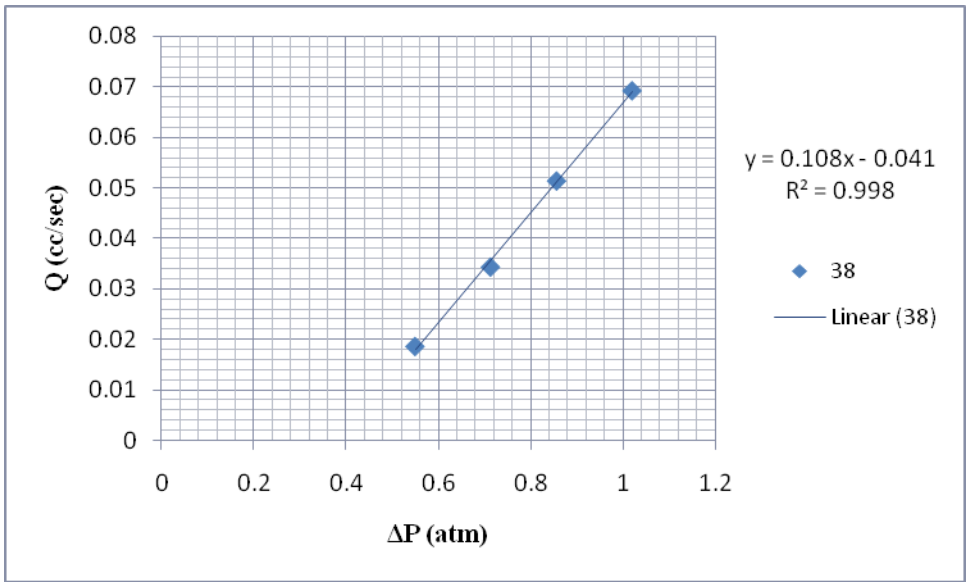

Figure B.1: Permeability calculation

$$K(md) = slope * \frac{\mu * L * 10^3}{A}$$

K is specific or absolute permeability in millidarcy

μ is dynamic viscosity of brine = 1.08112cp

L is length of sample in cm

A is cross sectional area of sample in sq.cm

From the absolute permeability, k (m/s) can be calculated from the relationship below

$$k = \frac{\gamma}{\mu} K$$

where

k is permeability m/s

γ is specific gravity of brine = $\rho_f g$ =1.0363×9.80665×1000 N/m^3

γ = 10162.63 N/m^3

μ is viscosity of brine in centipoise(cp) = 1.08112 cp

1 cp = 0.001 Pa.s

K is specific or absolute permeability in Darcy

1 Darcy = $9.869233 \times 10^{-13} m^2$

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